ABSTRACT

The U.S. Department of Energy’s Generation IV Program has generated considerable interest for High-Temperature Gas-Cooled Reactors (HTGR), in particular, the Very-High-Temperature Reactor (VHTR). VHTR is one of the six reactor concepts selected by the Generation IV International Forum and is anticipated to be reactor type for the Next Generation Nuclear Plant (NGNP). The VHTR concept, with a projected plant design service life of 60 years, is being actively researched not only due to its near-term deployment potential but also because it offers a broad range of process heat applications ranging from electricity generation to hydrogen co-generation. To efficiently and reliably transfer the thermal output from the reactor core, VHTRs require high temperature (900-950 °C) and high integrity heat exchangers with high effectiveness during normal and off-normal conditions. A class of compact plate-type heat exchangers, namely, Printed Circuit Heat Exchangers (PCHEs), made of high-temperature materials and found to have these above characteristics are being increasingly pursued for heavy duty applications.

The current thesis work is a part of a larger research project aimed at investigating the design, fabrication, testing, modeling, and optimization of PCHEs at operating temperatures proposed to be realized in VHTRs. In the present work, two PCHEs were designed and fabricated. In addition, a detailed design of a high-temperature helium test
facility to test the thermal-hydraulic performance of these PCHEs was completed. Owing to the high operating temperatures and pressures, a detailed investigation on various high-temperature materials was carried out to aid in the design of the test facility and the heat exchangers. The study showed that Alloys 617 and 230 are the leading candidate materials for high-temperature applications. However, economics and material availability in the required form dictated the final design operating conditions. The helium test facility is of Alloy 800HT construction and is currently being constructed at The Ohio State University. It is intended to facilitate experiments at operating temperatures and pressures up to 900 °C and 3 MPa, respectively. Two PCHEs, each having 10 hot and 10 cold plates with 12 channels in each plate, were fabricated using Alloy 617 plates. In addition, the processes related to the fabrication of PCHEs, namely photochemical machining and diffusion bonding techniques were investigated and successfully established for Alloy 617 plates. The PCHEs will be tested in the helium test facility for their heat transfer and pressure drop characteristics at various operating temperatures and pressures. The experimental database generated from the test facility will then be used for model development and computer code validation. In summary, the current thesis focuses on the study of the high-temperature materials, design of the helium test facility, design, development, and fabrication of the high-temperature PCHEs.
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CHAPTER 1

INTRODUCTION

1.1 Motivation

The U.S. Department of Energy’s Generation IV Program has generated considerable interest for High-Temperature Gas-Cooled Reactors (HTGR), in particular, the Very-High-Temperature Reactor (VHTR). VHTR is one of the six reactor concepts selected by the Generation IV International Forum and is anticipated to be the Next Generation Nuclear Plant (NGNP). The VHTR concept, with a projected plant design service life of 60 years, is being actively researched not only due to its near-term deployment potential but also because it offers a broad range of process heat applications ranging from electricity generation to hydrogen co-generation. Figure 1.1 shows a conceptual layout of the VHTR illustrating the nuclear heat source and the associated power conversion unit and the hydrogen generation plant [1]. The reactor design is a graphite-moderated, helium-cooled, prismatic or pebble-bed core, thermal neutron spectrum reactor with a once-through uranium fuel cycle and core outlet temperatures of 850-950 °C.

For realizing higher net plant efficiencies of around 50% or for efficient generation of hydrogen, it is necessary to efficiently and reliably transfer the thermal output from the core to the power conversion unit or to the hydrogen generation plant. In the VHTR
concept with indirect cycle, an intermediate heat exchanger (IHX) is required to transfer the thermal output from the core to the power generation unit and/or the hydrogen generation plant or other applications that require high process heat. In other words, VHTRs invariably require high-temperature, high-integrity, and high-efficiency heat exchangers in the primary coolant circuit as recuperators, or as an IHX for hydrogen production or in an indirect cycle for electricity generation. Despite numerous potential IHX concepts for VHTRs, it is generally agreed that Printed Circuit Heat Exchanger (PCHE) offers the most promising choice considering the extremity of the VHTR operating conditions and the inherent safety advantages it offers. PCHE is being actively researched for VHTR applications due to its compactness, high effectiveness and high mechanical integrity. It can also provide enhanced safety by removing decay heat during off-normal conditions. The design, fabrication, testing, and modeling of this key component for Generation IV reactor conditions are the principal objectives of our research.

Figure 1.1  The Very High-temperature Reactor [1]
1.2 Intermediate Heat Exchanger

Heat exchanger is one of the key components of the NGNP system. The purpose of IHX, as mentioned before, is to efficiently and reliably transfer the heat generated in the core to the power conversion system and the hydrogen generation plant. Conventional shell and tube heat exchangers cannot meet the space requirements either as a recuperator in the direct cycle or as an IHX in the indirect cycle. A most promising compact heat exchanger design, namely, the Printed Circuit Heat Exchanger is being actively researched for potential applications in VHTRs due to its compactness, high-effectiveness and high-integrity. PCHEs provide a compact alternative to shell and tube heat exchangers in that they are characterized by a large heat transfer area per unit volume of the exchanger.

PCHE is a compact plate-type heat exchanger in which fluid flow channels are photochemically etched on flat metal plates. The etched plates are then stacked together in a particular configuration and diffusion bonded together to form a heat exchanger block. Diffusion bonding is a solid state joining process in which the plate stack is subjected to thermal soaking for a certain period of time allowing for grain growth and thus enabling an interface-free joint between the plates. This process offers parent metal strength and very high pressure containment capability to the diffusion bonded block. Due to the inherent advantages of a diffusion bonded heat exchanger over a brazed one, PCHEs are being actively investigated for VHTRs and other Generation IV reactor systems. In the VHTR concept with an indirect cycle, an IHX is required to transfer the thermal output from primary helium from the core to the secondary fluid, which can be helium, a nitrogen/helium mixture, or a molten salt.
The operating conditions of the VHTRs represent a major departure from the existing light-water cooled reactor technologies. The IHXs of the VHTRs are required to withstand high temperatures (900-950 °C) for long duration of service without any considerable degradation of material mechanical properties and corrosion resistance ability. In addition, safety constraints preclude the integration of hydrogen generation plant with the nuclear power generation unit. Therefore, it becomes necessary to transfer the heat efficiently and reliably over relatively long distances without loss of mechanical integrity of the materials of the heat transport system. Material integrity at high temperatures is a key to the performance capability of a high-temperature heat exchanger.

Despite a significant assortment of materials and alloys for use in high-temperature applications, such as petrochemical, aerospace and power generation industries, very few have been tested or qualified for nuclear-related applications. As a part of the current thesis research, eight high-temperature materials were reviewed for their high-temperature mechanical properties (creep and creep-fatigue properties), physical properties (thermal conductivity and thermal expansion coefficient), environmental resistance, fabrication and joining technology (i.e., workability and weldability), availability and cost. This investigation, carried out at The Ohio State University, is primarily intended for identifying an appropriate high-temperature material for its high-temperature helium test facility piping and for the diffusion bonded compact PCHEs. The aim of the current thesis research is to design and build a high-temperature helium test facility and fabricate PCHEs for potential applications in VHTRs.
1.3 Previous Work

In the literature, some research work was performed with the PCHEs. Nikitin et al. and Ishizuka et al. [2, 3] investigated, both numerically and experimentally, the thermal-hydraulic performance of a 3-kW PCHE in a supercritical CO$_2$ loop with mass flow rates varying from 40-80 kg/h. In their experiments, the hot and cold side inlet temperatures were varied from 280-300 °C and 90-108 °C, respectively while the hot and cold side inlet pressures were varied from 2.2-3.2 MPa and 6.5-10.5 MPa, respectively. For the PCHE tested, they correlated local heat transfer coefficient and pressure drop in terms of Reynolds number. As is clearly evident, their experimental operating temperatures only reached 300 °C and are far from high-temperature reactor (HTR) requirements. Recently, Pra et al. [4] carried out steady and transient tests on a PCHE recuperator mock-up and investigated their thermal-mechanical behavior in an air test loop for conditions typical of High Temperature Reactor (510 °C). Their results showed that PCHE was a potential candidate for high temperature applications. Notwithstanding the above few works on PCHEs, to the authors’ knowledge, however, a detailed thermal hydraulic performance of the PCHEs with helium as the working fluid has not been investigated for the temperatures and pressures typical of VHTRs. Besides, there is essentially no experimental data available for the performance of a PCHE when the operating conditions deviate from the nominal design condition. This type of experimental data is critical to assess the safety performance of the entire reactor system under a transient or during a license review process. Furthermore, the U.S. fabrication capability of PCHEs has not been fully explored and confirmed. The current thesis research work addresses some of these issues and is in fact a part of the larger objective aimed at building an extensive database of the thermal-hydraulic characteristics of PCHEs under VHTR operating conditions.
1.4 Thesis Objectives

The objectives of the current thesis research are as follows:

a) To design and fabricate high-temperature PCHEs having potential applications in the VHTRs.

b) To investigate and identify appropriate high-temperature materials for the PCHEs and helium test facility.

c) To review the processes and explore the domestic capabilities related to the fabrication of PCHEs, such as the photochemical machining and diffusion bonding techniques, which in turn affects the design of PCHEs.

d) To confirm the applicability of the photochemical machining and diffusion bonding processes for high-temperature alloys such as Alloy 617.

e) To design and construct a high-temperature helium test facility and evaluate the thermal-hydraulic performance of the PCHEs under various conditions.

1.5 Thesis Organization

This thesis is comprised of six chapters and four appendices.

Chapter 1 introduces the problem statement, lays down the objectives of the current thesis research work, and provides background information on the need for a high-temperature, high-integrity and high-effectiveness compact heat exchanger.

Chapter 2 introduces the PCHEs and provides background information on the PCHEs. The processes related to the design and fabrication the PCHEs, such as the photochemical machining and diffusion bonding techniques are explained in detail. In addition, this chapter also covers the design and fabrication aspects of two high-temperature PCHE blocks manufactured from Alloy 617 plates. These two PCHEs will
be tested in the high-temperature helium test facility for their thermal-hydraulic performance under conditions typical of VHTR.

Chapter 3 details the literature survey on various high-temperature materials from the perspective of Generation IV reactor applications. This literature survey was primarily intended for selecting an appropriate high-temperature material for the heat exchangers and the helium test facility at The Ohio State University. Various high-temperature materials were investigated for their high-temperature strength, corrosion resistance and fabricability. Based on economics and availability, Alloy 617 was selected as the plate material for the PCHEs and Alloy 800HT for the piping of the high-temperature helium test facility, as well as the headers of the PCHEs.

Chapter 4 discusses the design aspects of the high-temperature helium test facility. This test facility is designed to facilitate operation at temperatures and pressures up to 900 °C and 3 MPa, respectively. Some major loop components, such as the electric heaters, the cooler, and the gas boosters are also discussed.

Chapter 5 introduces and discusses various heat transfer and pressure drop correlations that are applicable for semi-circular channels that are used for the PCHEs. The correlations cover both the laminar, turbulent, and laminar-to-turbulent transition flow regimes.

Chapter 6 summarizes the important results and findings of this thesis research and provides recommendations for future work, which is currently ongoing.

Appendices detailing the thermal-physical properties of helium (Appendix A), ASME allowable stresses for various high-temperature materials (Appendix B) and chromatographic analysis certificate of helium gas (Appendix C) follow.
CHAPTER 2

PRINTED CIRCUIT HEAT EXCHANGER

2.1 Introduction

For the VHTRs, it is essential to have reliable, efficient heat exchangers that can be operated at high-temperature, high-pressure conditions comparable to the VHTR operating conditions. The objective of the current research, broadly, has been to design, fabricate, test, and optimize the performance of PCHEs for potential applications in VHTRs. However, this thesis restricts itself only to the design and fabrication aspects of the research. In the following sections, a qualitative description of the PCHE along with its design, construction, and fabrication is presented. This discussion is motivated by the fact that PCHE is a relatively new concept and the subject matter of the present thesis research. The PCHE design was originally developed for refrigeration applications in 1985 and since then, only Heatric™ has been involved in the commercial design and manufacturing of this type of heat exchangers. In addition, the literature on these heat exchangers is very meager and their thermal-hydraulic performance data are essentially non-existent for temperatures typical of VHTRs. The design and fabrication of two high-temperature PCHEs using Alloy 617 plates forms the nucleus of this chapter. A broad overview of the photochemical machining and diffusion bonding techniques employed during the fabrication of these PCHEs is also presented.
2.2 PCHE Background Information

Printed Circuit Heat Exchanger, as the name implies, derives its name from a technique employed for the production of copper electronic printed circuit boards and silicon integrated circuits. It belongs to the category of compact heat exchangers as its heat transfer surface area density, a measure of heat exchanger compactness, is typically greater than 2,500 m\(^2\)/m\(^3\). A PCHE consists of flat metal plates in which fluid flow passages are photo-chemically milled on one side of each plate in much the same way as printed circuit boards. The plates are then stacked in a particular configuration and diffusion bonded together to form a block. Diffusion bonding is a solid state joining process in which an interface-free joint between the plates can be realized. Besides very high pressure containment capability, this procedure offers parent metal strength and ductility. The fluid flow passages have approximately a semi-circular cross section, being typically 0.5-2.0 mm wide. The passage shape may be corrugated or straight, depending on a number of factors, such as the fluid used, the heat load, and the pressure drop requirements. Depending on the heat duty requirements many diffusion-bonded blocks are face-welded to form a larger core. Headers and nozzles are welded directly onto the final core block.

PCHEs have several advantages over the conventional shell and tube heat exchangers as well as other compact heat exchangers like the brazed plate heat exchangers. They differ from brazed plate heat exchangers in that they employ diffusion bonding instead of brazing. PCHEs are typically four to six times smaller than the conventional shell and tube heat exchangers of equivalent duty and possess high pressure and temperature capability in excess of 50 MPa and 800 °C, respectively. Furthermore, the processes employed in PCHE fabrication, namely photochemical
machining and diffusion bonding are compatible with a wide range of materials, and this therefore allows them to be used for corrosive environments.

Figure 2.1 shows two different arrangements of plates. The plates can be arranged in a single or a double banking configuration. In the single banking configuration, hot and cold plates alternate each other while in a double banking configuration one fluid channel is sandwiched between two others.

Figure 2.1  PCHE: single and double banking configurations
2.3 Photochemical Machining

Photochemical machining (PCM) is one of the oldest and least known non-conventional machining processes. It is also known as photoetching, photochemical milling, chemical blanking and photofabrication. The origins of this process dates back as early as 2500 B.C. when ancient Egyptians employed it to produce copper jewellery etched with citric acid. Nowadays, this process is widely used to fabricate components used in micro-engineering, medical, electronic, aerospace and other industries. Currently, it is drawing considerable attention in the nuclear community for its applicability in the manufacture of high-temperature compact heat exchangers, namely, PCHEs. The fabrication of PCHEs involves producing small size (on the order of 2 mm) flow channels by photochemical machining on flat metal plates of high-temperature resistant materials, such as alloy 617.

Photochemical machining uses strong chemical solution to remove unwanted workpiece material by controlled dissolution [4]. In other words, it uses chemical etchants to corrosively oxidize selected areas of metal. Besides low cost, the advantages of this method include fast processing, no burring on the edges of the walls, processing of very thin materials, precise control of the channel dimensions and a short lead time between prototyping and production [5, 6]. Furthermore, it does not alter the internal structure of the metal and does not affect the metal properties like hardness, grain structure and ductility. However, this process has a limitation in that the etching is isotropic whereby the etchant will attack not only downwards into the metal but also sideways beneath the photoresist stencil layer [7]. Hence the depth of the channel is always less than the width of the channel. The ratio of etched depth to the undercut (or
lateral etch) is termed the etch factor and is illustrated in Figure 2.2. The undercut $U$ and etch factor are defined as [7]

$$U = \frac{(B - A)}{2}$$

(2.1)

Etch factor = \frac{D'}{U} \quad (2.2)

where $D$ is the depth of the channel, $A$ is the initial width of the photoresist opening on the substrate and $B$ is the final width of the channel at the top. High etch factor is generally desired.

![Figure 2.2 Etch factor](image)

2.3.1 The PCM Process

Before describing the steps involved in the PCM technique, it is important and worthwhile to understand some common terminology employed in photochemical machining processes [8].
a) Photoresist: A material which, when properly applied to any of a variety of substrates, becomes sensitive to portions of the electromagnetic spectrum and, when properly exposed and developed, masks portions of the substrate with a high degree of integrity.

b) Phototool: The working tool that is used in production for exposing the resist coated workpiece. It usually comprises of arrays of images on photographic film or plate (glass).

c) Photoplotter: Photoplotters produce phototools by exposing photographic film or plate with a beam of light, the shape, size, intensity and movement of which is controlled by a computer.

Figure 2.3 describes the multi-stage photochemical machining process [7]. This process mainly involves the generation of the artwork and the production of the phototool. Phototools are generally produced using CAD design and laser photoplotting by selectively exposing a photographic film. It is followed by cleaning the metal substrate to remove all dirt, rust, greases, and oils. The metal substrate is then sensitized by applying photoresist coating onto its surface. Photoresists are UV-light sensitive polymers and form a vital element of the PCM process. With the aid of a phototool, the sensitized metal substrate is then exposed to UV-light forming an adherent, durable image in the photoresist. The metal substrate with the photoresist stencil on it is then etched with a suitable chemical etchant. The photoresist is then stripped off from the etched substrate to leave the final burr- and stress-free product.
Figure 2.3 Flowchart of a typical photoetching process [7]
2.3.2 Photoetching of Alloy 617 Plates

Etching involves a heterogeneous chemical reaction in which etchant liquid reacts with a solid metal substrate and oxidizes it to produce a soluble reaction product [9]. An ideal etchant solution for any metal etching process should be stable, have a high etch rate, produce minimum undercut, and have high dissolved metal capacity [4, 10]. Despite a plethora of industrial etchants, the most commonly and widely used industrial etchant is aqueous ferric chloride solution as it is inexpensive and attacks a wide range of metals. In what follows, the basic chemistry behind etching of nickel superalloys is presented. However, it is to be noted that a number of other equally important processes like solvent cleaning the substrate, application of chemical coatings, etc., precede the etching process.

The chemical etchant typically used for etching alloy 617 plates is an aqueous mixture of ferric chloride (FeCl$_3$), hydrochloric acid (HCl), and nitric acid (HNO$_3$) [7]. Another potentially dangerous etchant, namely aqua regia (3 vol. HCl + 1 vol. HNO$_3$) is also employed for etching alloy 617 plates. The basic chemistry involved in etching of metals with a solution of FeCl$_3$ alone can be illustrated as follows:

$$n\text{FeCl}_3 + \text{M} \rightarrow n\text{FeCl}_2 + \text{MCl}_n$$  \hspace{1cm} (2.3)

where M denotes the metal being etched and n its valency [7]. As etching proceeds, the ferric ion is consumed and ferrous ion concentration increases and is deposited on the metal being etched, which slows down the rate of etching. Maintaining a constant etchant composition is vital for the etching process. Hydrochloric acid is added to improve the solubility of ferrous ion in the etchant solution and increase the rate of
etching. HNO$_3$ is used for obtaining smooth and uniform channels. It is economical to regenerate ferric chloride from the waste ferrous chloride byproducts by various methods of regeneration.

PCM process can be used for etching a wide range of materials, although some are more etch-friendly than others. As mentioned before, etching is basically rapid controlled corrosion. Corrosion-resistant materials are difficult to etch and require corrosive etchants. Alloy 617, the high-temperature material employed in the PCHEs being fabricated at The Ohio State University, has relatively high chromium and nickel contents and exhibits excellent resistance to a wide range of corrosive environments, which makes it difficult to etch. Therefore, to successfully etch alloy 617, corrosive etchants like the ones mentioned before are required. During this study, the etching of alloy 617 has been successfully established by PCM Products Inc. Figure 2.4 shows a photochemically etched alloy 617 plate.

![Alloy 617](image)

Figure 2.4 Photochemically etched alloy 617 plate
2.3.3 Component Dimensional Limitations of Etching

The size range of the parts that can be fabricated by PCM is limited by the ability of the PCM machine to handle large substrates. Currently, the maximum thickness of the material etched, for most PCM vendors in the United States, is around 2.5 mm and the maximum width of the photosensitive film necessary for etching process is limited to 600 mm. This puts a constraint on the maximum width of a single diffusion bonded heat exchanger module. However, depending on the heat duty requirements, many modules can be face-welded to form a larger heat exchanger core.

2.4 Diffusion Bonding

Solid-state diffusion bonding is a high-quality joining technique and can be classified broadly under the category of pressure welding. It is increasingly being employed for joining in heavy duty applications as an attractive alternative to conventional welding and brazing techniques. Currently, this process is drawing considerable interest in the nuclear engineering community for its applicability in the fabrication of printed circuit heat exchangers. As the name implies, the principal mechanism in solid-state diffusion bonding process is interdiffusion of atoms across the interface. The International Institute of Welding (IIW) has adopted a modified definition of solid-state diffusion bonding proposed by Kazakov [11]: “Diffusion bonding of materials in solid state is a process for making a monolithic joint through the formation of bonds at the atomic level, as a result of the closure of the mating surfaces due to local plastic deformation at elevated temperature which aids interdiffusion at the surface layers of the materials being joined.” This technique is also commonly referred to as pressure joining, diffusion welding, hot press bonding, auto-vacuum welding, thermo-compression welding, isostatic bonding, and solid-state or solid-phase welding.
Diffusion bonding is carried out in a bonding chamber in which vacuum is maintained. The materials to be bonded are placed in this chamber with their mating surfaces touching and heated to a specified temperature. A pressing load is then applied for a specified amount of time interval. After the joint is complete, the weldment is held in the chamber or in the air for cooling, depending on the properties of the materials involved.

The success or failure of diffusion bonding is governed by three bonding variables, namely, the bonding temperature, the bonding pressure (or pressing load), and the holding time (duration of pressure). With properly selected bonding variables, this process allows to realize the same joint strength and ductility equivalent to that of the parent metal. Diffusion bonding of most metals is conducted in vacuum (typically, 0.1-0.001 Pa) or in an inert atmosphere (normally dry nitrogen, argon or helium) in order to reduce detrimental oxidation of the faying surfaces. A very important distinction of this process relative to other joining processes is its high quality of joints, which ensures no metallurgical discontinuities and porosity across the interface. It can be used to join similar as well as dissimilar metals and alloys widely differing in thermo-physical characteristics. Diffusion bonding of similar materials under optimal conditions leaves no physical interface whatsoever between the pieces being joined while diffusion bonding of dissimilar materials can produce transition layers or new phases consisting of the constituents of the parent materials [11]. Table 2.1 outlines some of the advantages of diffusion bonding technique over conventional brazing technique.
Table 2.1  Comparison of diffusion bonding and brazing [11]

<table>
<thead>
<tr>
<th>Particulars</th>
<th>Diffusion Bonding</th>
<th>Brazing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Contacting method</td>
<td>Pressure</td>
<td>Contact fusion, contact fusion and</td>
</tr>
<tr>
<td></td>
<td></td>
<td>pressure, pressure and contact fusion</td>
</tr>
<tr>
<td>Bonding</td>
<td>Adhesive, diffusion</td>
<td>Cohesive, adhesive</td>
</tr>
<tr>
<td>Heating</td>
<td>Local, total</td>
<td>Local, total</td>
</tr>
<tr>
<td>Temperature</td>
<td>50-80% of melting point of parent metal</td>
<td>Somewhat above melting point of solder</td>
</tr>
<tr>
<td>Surface preparation</td>
<td>Careful</td>
<td>Less exacting</td>
</tr>
<tr>
<td>Materials</td>
<td>Metals, alloys, nonmetals</td>
<td>Metals, alloys, nonmetals</td>
</tr>
<tr>
<td>Porosity</td>
<td>None</td>
<td>Blowholes, shrinkage, diffusion</td>
</tr>
<tr>
<td>Vibration survival</td>
<td>Very high</td>
<td>High</td>
</tr>
<tr>
<td>Corrosion resistance</td>
<td>Fairly high</td>
<td>Low</td>
</tr>
<tr>
<td>Strength</td>
<td>That of parent metal</td>
<td>That of solder</td>
</tr>
</tbody>
</table>

The following requirements are to be invariably met for producing a high-quality diffusion bond [11].
a) The mating surfaces must be thoroughly cleaned and a direct contact between them ensured.

b) The materials should be heated to a temperature promoting diffusion across the mating surfaces over a specified time interval.

c) The atmosphere around the joint should shield the material against oxidation, or contamination or both.

2.4.1 Principal Bonding Variables

As mentioned before, the quality of the diffusion bonded joint is a strong function of four interrelated variables, namely, the bonding temperature, the bonding pressure, the vacuum in the process chamber, and the holding time (duration of pressure). To describe and understand the relationships among these variables would be a thesis in itself. Interested reader may refer to Kazakhov [11] to get an in-depth appreciation of these variables and the relationships among them. The present discussion, however, attempts to focus on and restricts itself to only a concise description of these variables.

a) Bonding Temperature: The bonding temperature typically ranges between 50% and 80% of the melting point of the most fusible metal in the composition. It is chosen such that plastic deformation (crushing of surface asperities) and diffusion processes (interdiffusion of atoms across the interface of the weld) occur at a sufficiently high rate.

b) Bonding Pressure: Bonding pressure or pressing load is primarily intended to ensure a tight contact between the mating surfaces, to break up and disperse the brittle surface oxide films, to aid in the deformation of surface asperities and fill the voids in the weld zone. It activates recrystallization and promotes interdiffusion of atoms across the interface of the materials being joined. An
optimal bonding pressure, typically chosen equal to the yield strength of the materials being joined at the bonding temperature, is one that produces a sound weld and does not lead to macroscopic deformation in the weld zone.

c) Holding time: Holding time at a specified bonding temperature and a specified bonding pressure should be just sufficient for an intimate contact to be formed and for diffusion processes to take place. An excessive diffusion time might leave voids in the weld zone or even change the chemical composition of the metals being joined.

d) Vacuum: Vacuum is maintained in the bonding chamber to provide sufficient rarefaction and protect the workpieces against intensive oxidation as they are heated. When the workpiece is heated in vacuum, its oxides, nitrides, and hydrides are caused to dissociate and vaporize. Thus, vacuum serves to degas the materials being joined and help in removal of oxides and other impurities from both the surface and the bulk of the materials. Typically, a vacuum pressure of 0.1 to 0.001 Pa is maintained in the bonding chamber. It has been observed that diffusion bonded joints made in a vacuum of 13.3 Pa are better than those made in especially pure argon carrying 0.003% O₂ and 0.003% N₂ [11].

It is essential to understand the relationships among the above variables to operate under optimal conditions for realizing high-quality diffusion bonded joint.

2.4.2 Mechanism of Bond Formation

If ideal metallic specimen surfaces, i.e., smooth, clean and parallel surfaces are brought in contact, metallic bonds sufficient for a joint to form would be established spontaneously without any external energy input. However, in practice, all real metal specimen surfaces are rough with a multitude of microscopic asperities and are covered
by oxide films, adsorbed grease and gases which interfere with the formation of metallic bonds. In other words, formation of metallic bonds between the real specimen surfaces involves a sequence of steps. There are a multitude of hypotheses to explain the bond formation mechanism in solid-state diffusion bonding process. However, to date, no single hypothesis has been successful in offering a complete explanation for the mechanism of bond formation. A discussion on all these hypotheses is unwarranted and beyond the scope of this thesis research. Nevertheless, for the ease of understanding, the mechanism of solid-state diffusion bonding can be understood by breaking up the operation into two steps. Figure 2.5 illustrates the two stages involved in solid-state diffusion bonding [12]. The first stage involves plastic deformation of asperities on the mating surfaces as the pressure is applied. This microplastic deformation proceeds until the localised effective stress at the contact area becomes less than the yield strength of the material at the bonding temperature. In fact, initial contact occurs between the oxide layers that cover the faying surfaces. As the deformation of asperities proceeds, more metal-to-metal contact is established because of local disruption of the relatively brittle oxide films which generally fracture readily. At the end of the first stage, the bonded area is less than 10% and a large volume of voids and oxide remains between localised bonded regions. The second stage of bonding is characterized by thermally activated mechanisms, namely creep and diffusion, which lead to void shrinkage and increase of the bonded area.
2.5 PCHE Design and Fabrication

PCHE, as mentioned before, is formed by diffusion bonding a stack of flat plates with flow passages photochemically etched on them. Two PCHEs were designed and fabricated at The Ohio State University to test their thermal-hydraulic performance at temperatures and pressures up to 900 °C and 3 MPa, respectively. For reasons discussed in Chapter 3, Alloy 617 was selected as the plate material for the heat exchangers. However, as received Alloy 617 plates do not yield a high quality diffusion bond joint due to the inherent presence of oxide films on the plate surface. Aluminum and Titanium (highly reactive materials) in Alloy 617 form protective oxides on the surface. The protective oxides on the plate surface were electrochemically removed and a very thin layer \((10^{-4}-1.5\times10^{-4} \text{ inch})\) of pure nickel is electrolytically deposited on the plate surface to prevent any further oxidation. This Ni interlayer facilitates diffusion
bonding. Figure 2.6 shows two diffusion bonded alloy 617 heat exchanger cores. Proof pressure testing with helium on the diffusion bonded Alloy 617 PCHE core was performed and the results confirmed that the bonds were leak tight. Besides, the diffusion bonded block is found to have a very high pressure containment capability.

![Two diffusion bonded Alloy 617 heat exchanger cores](image)

**Figure 2.6** Two diffusion bonded Alloy 617 heat exchanger cores

### 2.5.1 PCHE Design

In what follows, the design, construction, and fabrication of the PCHEs are presented. Two PCHEs were designed and fabricated from Alloy 617 plates to evaluate their thermal-hydraulic performance in a high-temperature helium test facility. The geometric details of both the PCHEs are tabulated in Table 2.2. The fluid flow passages are photochemically etched on flat plates of Alloy 617. The plates are then stacked in a single banking configuration with hot and cold plates alternating each other and diffusion bonded together to form a heat exchanger core. Each PCHE has core dimensions of $70.7 \times 101.6 \times 304.8$ mm$^3$. The fluid flow channel passages are approximately semi-circular in cross-section with a width of 2 mm and a pitch of 2.5 mm. In the current design, the shape of the flow passage may be described as straight. Except for the cross
flow at entry and exit regions of each side of the PCHE, all the active heat transfer passages are in pure counter-current flow. Each PCHE has 10 hot and 10 cold plates with 12 straight channels in each plate. The number of plates and the number of channels in each plate were dictated by maximum mass flow rate of 80 kg/h in the test facility. The current PCHE configuration allows it to be operated in the laminar and laminar-to-turbulent transition regimes. Figures 2.7 and 2.8 show the plate design, channel cross-section details, header design, and the complete assembly of the PCHE. It is to be noted that the headers that will be eventually welded on to the PCHE are fabricated using Alloy 800HT solid rods.

Table 2.2  PCHE geometric details

<table>
<thead>
<tr>
<th></th>
<th>Hot Side</th>
<th>Cold Side</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plate dimension (mm)</td>
<td>$101.6 \times 304.8$</td>
<td>$101.6 \times 304.8$</td>
</tr>
<tr>
<td>Plate thickness (mm)</td>
<td>1.63</td>
<td>1.63</td>
</tr>
<tr>
<td>Number of plates</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Number of channels in each plate</td>
<td>12</td>
<td>12</td>
</tr>
<tr>
<td>Channel pitch (mm)</td>
<td>2.5</td>
<td>2.5</td>
</tr>
<tr>
<td>Channel width (mm)</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Channel depth (mm)</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Channel hydraulic diameter (mm)</td>
<td>1.22</td>
<td>1.22</td>
</tr>
<tr>
<td>Channel travel length (mm)</td>
<td>304.8</td>
<td>271.5</td>
</tr>
<tr>
<td>Heat transfer area, (m²)</td>
<td>0.188</td>
<td>0.168</td>
</tr>
<tr>
<td>Channel c/s area, (mm²)</td>
<td>1.57</td>
<td>1.57</td>
</tr>
<tr>
<td>Free flow area, (mm²)</td>
<td>188</td>
<td>188</td>
</tr>
</tbody>
</table>
Figure 2.7  PCHE plate dimensions (in inches) (a) straight pattern, and (b) z pattern
2.5.2 PCHE Fabrication

As mentioned before, two PCHE cores were fabricated using Alloy 617 plates. Figures 2.9(a) and (b) show Alloy 617 plates with two different flow patterns.
photochemically etched on them. It is to be noted that the shape of the flow passage in both the cases is straight. Figure 2.9(c) shows the channel cross section, which is approximately semi-circular. Figures 2.10(a) and (b) show the diffusion bonded heat exchanger modules without headers.

Figure 2.9  Photchemically etched Alloy 617 plates (a) straight pattern, (b) z pattern, and (c) channel cross-section
Figure 2.10  Diffusion bonded Alloy 617 heat exchanger core (a) z pattern side, and (b) straight pattern side
Generally for PCHEs, the flow passage shape can be straight (non-corrugated) or wavy (corrugated or herringbone). However, in the current research, a simple design with straight channels was employed mainly for two reasons:

a) To gain insight into the processes related to the fabrication of PCHEs, namely photochemical machining and diffusion bonding using Alloy 617 plates.

b) Straight channel geometry is well understood and pressure drop and heat transfer correlations for different flow regimes are well established.

Subsequently, a more compact PCHE with chevron (wavy) channels will be designed and fabricated. A wavy channel design will improve the heat transfer performance significantly as compared to the conventional straight channels. This is because the flow in a wavy channel experiences entrance effects at each turn, which increases the heat transfer coefficient significantly. However, a wavy channel will result in longer flow paths and a much higher pressure drop. It is to be noted that the performance improvement realized with a wavy channel would be different for laminar and turbulent flow conditions. An optimum design of the wavy channels that provides an appreciable improvement in the heat transfer performance without significant penalty in pressure drop is being worked out.

2.5.3 PCHE Dimensional Limitations

a) The current fabrication constraints of a single module are 1500 mm × 600 mm on plate dimension and 600 mm on plate stack height. The maximum width is constrained by the width of the photosensitive film necessary for etching and it is currently 600 mm wide. However, depending on the heat duty requirements, many such modules can be face-welded to form a larger core.
2.6 Surface Roughness Measurement

Information of surface roughness of flow channels is required in the estimation of channel frictional pressure drop. Roughness on the surfaces causes local flow separation and reattachment. This increases the heat transfer coefficient as well as the friction factor. If the height of the roughness element is negligible compared to the flow cross section size, then roughness has little effect on laminar flow, whereas it exerts a strong influence on the turbulent flow.

The surface roughness of the flow channel interior was measured using a Veeco-Wyko NT3300 non-contact optical profilometer [3]. The profilometer was calibrated in Vertical Scanning Interferometry (VSI) mode and a 2.5× objective lens was used. In the framework of this technique, a substrate is illuminated by white or monochromatic light through a beam splitter. Image processing was carried out by means of Wyko Vision 32® software package. Figure 2.11 shows a three-dimensional image of the surface data of a representative channel interior surface. Table 2.3 lists the basic surface statistics of the same channel. Various surface roughness indicators, $R_a$, $R_q$, $R_t$, and $R_z$ represent average roughness, root mean square roughness, maximum height of the surface, and the average maximum height of the profile, respectively. The average surface roughness $R_a$ of the interior surface of the channel is less than one micrometer.

In our study, a total of 12 roughness measurements were made at random locations on three different channels, which resulted in an average roughness of 0.4 µm. As a comparison, Ishizuka et al. [4] measured an average surface roughness value of 2-3 µm in the flow channels of their diffusion bonded compact heat exchanger.
Figure 2.11 Three-dimensional contour map of the interior surface of the channel

Table 2.3 Surface roughness of the interior surface of a representative flow channel

<table>
<thead>
<tr>
<th>$Ra$ (nm)</th>
<th>$Rq$ (nm)</th>
<th>$Rt$ ($\mu$m)</th>
<th>$Rz$ ($\mu$m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>242.6</td>
<td>311.7</td>
<td>3.2</td>
<td>4.0</td>
</tr>
</tbody>
</table>
CHAPTER 3

REVIEW ON HIGH-TEMPERATURE MATERIALS

3.1 Introduction

There is a significant assortment of materials and alloys for use in high-temperature applications commonly encountered in petrochemical, aerospace and power generation industries. However, high-temperature reactor specific environment coupled with the proposed high operating temperatures and pressures calls for a judicious selection of the materials. A total of eight high-temperature materials were reviewed for their high-temperature mechanical properties (creep and creep-fatigue properties), physical properties (thermal conductivity and thermal expansion), reactor specific environmental resistance, fabricability, availability and economics. Although the current investigation was carried out as a part of material requirements for the high-temperature helium test facility being built at The Ohio State University, it will also serve to highlight some of the potential material issues for VHTR applications and benefit the NGNP project. This review may be exhaustive from the perspective of material requirements for the high-temperature helium test facility and the PCHEs. Nevertheless, it may well provide a good overview of the past and the current developments in the research on high-temperature materials for various high-temperature reactor programs. Interested reader may refer various references listed in this chapter to gain a better insight of the current developments in the high-temperature materials.
3.1.1 Metallurgical Terminology

In what follows, some commonly used metallurgical terminology is presented. It will serve to supplement the understanding of the material presented in this chapter.

a) Carburization: Refers to the process of adding carbon to a metal.

b) Grain: Metals are crystalline and in the solid state, the atoms of a metal or alloy have various crystallographic arrangements, like cubic or hexagonal structures. Unique crystalline aggregates of atoms are called grains. In an alloy, there are usually many grains with random orientation directions from grain to grain. The peripheral surface of a grain is called a grain boundary.

c) Grain Size: Grain size is a microstructural property of a material that indicates how large the crystals constituting the structure are. It is important for a number of physical and mechanical properties. For example, room temperature strength is increased by having a small grain size. High-temperature, long-term properties, such as creep, are improved by a large grain size. Therefore, a balance between the required properties is essential.

d) Oxidation: Refers to the corrosion of a metal that is exposed to an oxidizing gas at elevated temperatures. The long term resistance of structural alloys at high temperatures relies on the formation of a compact, adherent, and slow-growing surface oxide layer. If the oxide layer developed is not strong enough, then the impure helium will exchange carbon with the alloy material leading to changes in the alloy microstructure and the resulting mechanical properties [13].

e) Creep: Creep refers to progressive accumulation of plastic strain in a specimen under stress at elevated temperature over a period of time. Some typical creep limits for material applications are 1% in 10,000 hours (or a creep rate of
0.0001% per hour) for aircraft turbine parts and 1% in 100,000 hours (or a creep rate of 0.00001% per hour) for steam turbines and other similar equipment. A number of techniques exist for approximating long-term creep behavior based on a series of short-term creep tests. Larson-Miller theory postulates that for each combination of material and stress level, there exists a unique value of a parameter $P$ that is related to temperature and time by the equation,

$$P = (\theta + 460)(C + \log_{10} t)$$  

(3.1)

where

$P = \text{Larson-Miller parameter, constant for a given material and stress level}$

$\theta = \text{temperature, } ^\circ\text{F}$

$C = \text{constant, usually assumed to be 20}$

$t = \text{time in hours to rupture or to reach a specified value of creep strain}$

This equation was investigated for both creep and rupture for some 28 different materials by Larson and Miller with good success.

Table 3.1  Equivalent conditions based on Larson-Miller parameter

<table>
<thead>
<tr>
<th>Operating Condition</th>
<th>Equivalent Test Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>10,000 h at 1000 °F</td>
<td>13 h at 1200 °F</td>
</tr>
<tr>
<td>1,000 h at 1200 °F</td>
<td>12 h at 1350 °F</td>
</tr>
<tr>
<td>1,000 h at 1350 °F</td>
<td>12 h at 1500 °F</td>
</tr>
<tr>
<td>1,000 h at 300 °F</td>
<td>2.2 h at 400 °F</td>
</tr>
</tbody>
</table>
f) Fatigue: Fatigue failure is a sudden and catastrophic separation of a machine part into two or more pieces as a result of the application of fluctuating loads or deformations over a period of time. Failure takes place by the initiation and propagation of a crack until it becomes unstable and propagates suddenly to failure. Loads and deformations that cause failure by fatigue are far below the static or monotonic failure levels. Fatigue testing is done to evaluate a material's response to cyclic loading.

g) Low-Cycle Fatigue (LCF): When loads or deformations are of such a magnitude that less than about 10,000 cycles are required to produce failure, the phenomenon is usually termed low-cycle fatigue. This domain of cyclic loading is associated with high loads and short lives.

h) High-Cycle Fatigue (HCF): When loads or deformations are of such magnitude that more than about 10,000 cycles are required to produce failure, the phenomenon is usually termed high-cycle fatigue. This domain of cyclic loading is associated with lower loads and long lives.

i) Austenitic Stainless Steels: Stainless steels are iron-based alloys and can be martensitic (body centered tetragonal), austenitic (FCC), or ferritic (BCC) depending on the alloying additions to the iron. Austenitic stainless steels have high chromium and high nickel content and can be used up to 800 °C. Nickel is required to stabilize the gamma or face centered cubic (FCC) phase of the iron, and chromium imparts corrosion resistance. SS304 and SS316 are some common examples of austenitic stainless steels.

j) Ferrous materials: Ferrous materials are those that contain, by weight, more iron than any other single element; contain carbon content generally less than 2%, and contain other elements in the composition.
k) Nonferrous materials: Nonferrous materials are those that contain some other element in amounts more than any other element, including carbon.

l) Solution-Annealing: Solution-annealed material is in its most corrosion-resistant and ductile condition. In this process, the alloy material is heated to high temperatures (usually in air or some protective atmosphere or vacuum) to dissolve carbides into the solid austenitic phase and then cooled fast enough that the carbon does not have time to precipitate and grow into carbide phase. Solution refers to the microstructure.

3.2 High-temperature Resistant Materials

There is a significant body of work in the literature from late 1970s and early 1980s on various high-temperature resistant materials for applications in Gas-Cooled Reactors (GCRs). However, this past experience is not sufficient to qualify the metallic components in a Generation IV reactor like VHTR as the material environment (high-temperature) envisaged in VHTRs is different from the former GCRs. Besides, the materials being investigated currently are partly different from those previously investigated for GCRs. It is generally agreed that the key aspects that need to be addressed to guarantee the structural integrity of the metallic components are the microstructural stability, the creep strength, and compatibility with the coolant gas i.e., helium. Various high-temperature materials were investigated for their high-temperature mechanical properties (creep and creep-fatigue properties), physical properties (thermal conductivity and thermal expansion), environmental resistance, fabrication and joining technology, availability and economics. This exercise was carried out to arrive at an appropriate candidate material for the construction of the helium test facility and the heat exchangers for operating conditions typical of VHTR.
The candidate materials identified for review in the current study for use at design temperatures of 900 °C are,

- Alloy 617
- Alloy 230
- Alloy HX
- Incoloy Alloy 800HT (hereafter referred to as Alloy 800HT)
- Alloy 800H
- Alloy 625
- Alloy HR160
- Alloy 556

Table 3.2 lists the chemical composition (wt. %) of the eight high-temperature resistant alloys. All the alloys with the exception of 800H/HT are nickel based super alloys. Alloy 800H/HT is an iron-based super alloy. Table 3.3 lists the nominal chemical composition of these alloys along with their UNS (Unified Numbering Scheme) numbers and ASME specifications for seamless pipe. It is to be noted that Alloy 800HT has nearly identical chemical composition limits as Alloy 800H. The base elements in both alloys are the same. However, the alloys differ in the chemical composition limits of carbon, aluminum, and titanium. The C and Al + Ti contents for Alloy 800HT are restricted to 0.06-0.1 % and 0.85-1.2 %, respectively as opposed to 0.05-0.1 % and 0.3-1.2 % in Alloy 800H. This slight difference in chemical composition gives Alloy 800HT higher maximum allowable design stresses than Alloy 800H.
Table 3.2   Chemical composition (wt. %) specification of high-temperature alloys

<table>
<thead>
<tr>
<th></th>
<th>Alloy 617</th>
<th>Alloy 230</th>
<th>Alloy 800H</th>
<th>Hastelloy X</th>
<th>Alloy HR160</th>
<th>Alloy 556</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>44.5 min.</td>
<td>Balance</td>
<td>30.0-35.0</td>
<td>Balance</td>
<td>Balance</td>
<td>20.0</td>
</tr>
<tr>
<td>Cr</td>
<td>20.0-24.0</td>
<td>20.0-24.0</td>
<td>19.0-23.0</td>
<td>20.5-23.0</td>
<td>28.0</td>
<td>22.0</td>
</tr>
<tr>
<td>Mo</td>
<td>8.0-10.0</td>
<td>1.0-3.0</td>
<td>-</td>
<td>8.0-10.0</td>
<td>1.0 max.</td>
<td>3.0</td>
</tr>
<tr>
<td>Al</td>
<td>0.8-1.5</td>
<td>0.2-0.5</td>
<td>0.15-0.6</td>
<td>0.5 max.</td>
<td>-</td>
<td>0.2</td>
</tr>
<tr>
<td>C</td>
<td>0.05-0.15</td>
<td>0.05-0.15</td>
<td>0.05-0.1</td>
<td>0.05-0.15</td>
<td>0.05</td>
<td>0.10</td>
</tr>
<tr>
<td>Fe</td>
<td>3.0 max.</td>
<td>3.0 max.</td>
<td>39.5 min.</td>
<td>17.0-20.0</td>
<td>2.0 max.</td>
<td>Balance</td>
</tr>
<tr>
<td>Mn</td>
<td>1.0 max.</td>
<td>0.3-1.0</td>
<td>1.5</td>
<td>1.0 max.</td>
<td>0.5</td>
<td>1.0</td>
</tr>
<tr>
<td>Si</td>
<td>1.0 max.</td>
<td>0.25-0.75</td>
<td>1.0 max.</td>
<td>1.0</td>
<td>2.75</td>
<td>0.4</td>
</tr>
<tr>
<td>S, max.</td>
<td>0.015</td>
<td>0.015</td>
<td>0.015</td>
<td>0.03</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Ti</td>
<td>0.6 max.</td>
<td>-</td>
<td>0.15-0.6</td>
<td>0.15 max.</td>
<td>0.5</td>
<td>-</td>
</tr>
<tr>
<td>P, max.</td>
<td>0.03</td>
<td>0.03</td>
<td>-</td>
<td>0.04</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Others</td>
<td>Co: 10.0-15.0, B: 0.006 max., Cu: 0.5 max.</td>
<td>W: 13.0-15.0, Co: 5.0 max., B: 0.015 max., La: 0.005-0.05</td>
<td>Cu: 0.75</td>
<td>Co: 0.5-2.5, W: 0.2-1.0, Cu: 0.5 max., Ti</td>
<td>Co: 29.0, W: 1.0 max., Cb: 1.0 max., La: 0.02, Zr: 0.02, Ta: 0.6, N: 0.2, W: 0.5</td>
<td>Co: 18.0,</td>
</tr>
<tr>
<td>Heat Treatment</td>
<td>Solution annealed</td>
<td>Solution annealed</td>
<td>Solution annealed</td>
<td>Solution annealed</td>
<td>Solution annealed</td>
<td>Solution annealed</td>
</tr>
</tbody>
</table>
Table 3.3 Nominal Chemical Composition of High-Temperature Alloys [14]

<table>
<thead>
<tr>
<th>Alloy</th>
<th>UNS Number</th>
<th>Product Form</th>
<th>Spec No.</th>
<th>Nominal Chemical Composition (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>617</td>
<td>N06617</td>
<td>Seamless pipe &amp; tube</td>
<td>SB-167</td>
<td>52Ni-22Cr-13Co-9Mo</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Plate, sheet, strip</td>
<td>SB-168</td>
<td></td>
</tr>
<tr>
<td>230</td>
<td>N06230</td>
<td>Seamless pipe &amp; tube</td>
<td>SB-622</td>
<td>57Ni-22Cr-14W-2Mo-La</td>
</tr>
<tr>
<td>HR-160</td>
<td>N12160</td>
<td>Seamless pipe &amp; tube</td>
<td>SB-622</td>
<td>37Ni-30Co-28Cr-2.7Si</td>
</tr>
<tr>
<td>HX</td>
<td>N06002</td>
<td>Seamless pipe &amp; tube</td>
<td>SB-622</td>
<td>47Ni-22Cr-9M0-18Fe</td>
</tr>
<tr>
<td>556</td>
<td>R30556</td>
<td>Seamless pipe &amp; tube</td>
<td>SB-622</td>
<td>21Ni-30Fe-22Cr-18Co-3Mo-3W</td>
</tr>
<tr>
<td>800HT</td>
<td>N08811</td>
<td>Seamless pipe &amp; tube</td>
<td>SB-407</td>
<td>33Ni-42Fe-21Cr</td>
</tr>
<tr>
<td>800H</td>
<td>N08810</td>
<td>Seamless pipe &amp; tube</td>
<td>SB-407</td>
<td>33Ni-42Fe-21Cr</td>
</tr>
<tr>
<td>625</td>
<td>N06625</td>
<td>Seamless pipe &amp; tube</td>
<td>SB-444</td>
<td>60Ni-22Cr-9Mo-3.5Cb</td>
</tr>
</tbody>
</table>

3.3 ASME Codification and Allowable Design Stresses

The International Boiler and Pressure Vessel Code (B&PVC) of the American Society of Mechanical Engineers (ASME) establishes rules of safety governing the design, fabrication, and inspection of boilers and pressure vessels, and nuclear power plant components during construction [14]. For a material to be used for a particular application, it is necessary to gain acceptance from the ASME Boiler and Pressure Vessel Code. Section II of the ASME B&PVC addresses materials approved for use by the construction subcommittees. In addition to providing specifications for ferrous, nonferrous, and welding materials, Section II also provides tables of design stress
values, tensile and yield strength values, and material properties. Section II, Part D, Appendix 5 of the ASME B&PVC provides guidelines for new materials approval. NGNP’s higher temperatures and operating environment mandates significant effort for the approval of the potential candidate materials. For nuclear applications involving elevated temperature service, Section II approval must be followed by Section III, Subsection NH (Elevated Temperature Design) approval. Many preliminary NGNP candidate materials are not included in Subsection NH and Code Cases for potential NGNP candidate materials have to be developed. In fact, there are no materials approved to ASME III for temperatures approaching 900 °C. Currently, among the potential candidate materials, only Alloy 800H is approved by Section III Subsection NH for use up to 760 °C. Efforts are underway to modify Subsection NH to allow for increase in the maximum permitted temperatures and extend the allowable time-dependent stresses from the current 300,000 h to 600,000 h.

Tables 1A and 1B of Section II, Part D, of the ASME B&PV Code contain the maximum allowable stress values at various temperatures for ferrous and nonferrous materials, respectively. Table 3.4, reproduced in part here from Table 1-100 of Appendix 1 Section II Part D, establishes the criteria for allowable stress values from Tables 1A and 1B. The maximum allowable stress value shall be the lowest value obtained from the criteria in Table 1-100 (Table 3.4 here). In what follows, nomenclature of the terms used in Table 3.4 is given [14].

\[ F_{avg} = \text{multiplier applied to average stress for rupture in 100,000 h. At 1500 °F and below, } F_{avg} = 0.67. \text{ Above 1500 °F, it is determined from the slope of the log} \]
time-to-rupture versus log stress plot at 100,000 h such that \( \log F_{\text{avg}} = 1/n \), but it may not exceed 0.67

\[
R_T = \text{ratio of the average temperature dependent trend curve value of tensile strength to the room temperature tensile strength}
\]

\[
R_Y = \text{ratio of the average temperature dependent trend curve value of yield strength to the room temperature yield strength}
\]

\[
S_C = \text{average stress to produce a creep rate of 0.01 \% per 1000 h}
\]

\[
S_{\text{Ravg}} = \text{average stress to cause rupture at the end of 100,000 h}
\]

\[
S_{\text{Rmin}} = \text{minimum stress to cause rupture at the end of 100,000 h}
\]

\[
S_T = \text{specified minimum tensile strength at room temperature, ksi}
\]

\[
S_Y = \text{specified minimum yield strength at room temperature, ksi}
\]

In the application of these criteria, the yield strength at temperature is considered to be \( S_Y R_Y \), and the tensile strength at temperature to be \( 1.1 S_T R_T \)

**Table 3.4** Criteria for establishing allowable stress values for Tables 1A and 1B [14]
3.3.1 ASME Allowable Design Stresses for Potential Candidate Materials

The high-temperature helium test facility that is designed and being built at The Ohio State University is not intended for direct nuclear reactor applications itself. Instead, it is designed to facilitate experiments at operating temperatures and pressures up to 900 °C and 3 MPa, respectively for compact heat exchangers and other components testing. The material selection for the test facility is therefore primarily based on allowable stresses from Section II, Part D approved for ASME Section VIII, Division 1 construction.

Figure 3.1 compares the ASME allowable design stresses for a seamless pipe at different temperatures ranging from 300 to 1000 °C for the above candidate materials. It is to be noted that the allowable design stress values would be different for different product forms, i.e., the stress values depend on whether the product form is a seamless pipe or a welded pipe or a plate. However, for the aforementioned alloys, no difference in the allowable stress values was noticed between the seamless pipe and the plate form. The allowable design stress values in Figure 3.1 are obtained from Table 1B, Section II Part D of the ASME B&PVC and are applicable for both the product forms, namely seamless pipe and the plate. It is clear that only Alloys 617, 230, and 800H are approved for temperatures up to 982 °C. With the exception of Alloys HR160 and 625 which are approved for up to 815 and 871 °C, respectively, all other alloys are approved up to about 900 °C. Although Alloy 556 has higher allowable strength at 900 °C compared to all other alloys (except 617), its resistance in nitriding media is poor when compared to Alloys 617, 230 and HX. As will be evident from the remainder of this chapter, the current study (based on high-temperature strength, corrosion resistance, and fabricability) indicated that either alloy 617 or alloy 230 is the most suitable material for high-temperature and high-pressure (up to 7.0 MPa) applications. However, as will be
explained later, Alloy 617 was selected for the heat exchanger plates and Alloy 800HT for the test facility piping requirements.

![Graph showing maximum allowable design stresses for candidate materials](image)

**Figure 3.1** Maximum allowable design stresses for candidate materials

### 3.4 Discussion on Potential Candidate Materials

In what follows, a detailed discussion of only five high-temperature materials is presented. Among the alloys mentioned before, Alloys 625, HR160, and 556 are excluded from further discussion. The rationale behind this is that Alloys HR160 and 625 are allowed by ASME up to 815 and 871 °C, respectively, which are less than the required design temperature requirement of 900 °C for the test facility and the heat exchangers. Although, Alloy 556 is approved for Section VIII, Division 1 construction up to 900 °C, its resistance to nitriding environments is poor. Therefore, further discussions are restricted to only five high-temperature materials, i.e., Alloys 617, 230, HX, 800H, and 800HT.
3.4.1 Alloy 617

Alloy 617 (UNS N06617) is one of the leading candidates to meet the structural material requirements of the advanced gas-cooled reactors. It is a solid-solution strengthened, nickel-chromium-cobalt-molybdenum-aluminum alloy having an exceptional creep strength and oxidation resistance at temperatures above 870 °C. Nickel, chromium, and aluminum provide good cyclic oxidation and carburization resistance at high temperatures, while cobalt and molybdenum provide solid-solution strengthening. Its excellent creep-rupture strength even at elevated temperatures and its ability to retain toughness after long-time exposure at elevated temperatures makes it a potential candidate material for construction of the IHX for the NGNP. This alloy exhibits good metallurgical stability and good hot workability.

Allowable design stresses for alloy 617 products are found in Table 1B of Section II, Part D of the ASME B&PVC [14]. Alloy 617 products are solution-annealed as it provides a coarse grain structure and the best creep-rupture strength. It has good fabricability and lower thermal expansion than most other austenitic alloys. However, alloy 617 is susceptible to internal oxidation (related to aluminum and titanium contents) and radioactive contamination (Co≈10-15 wt. %). However, this may not be a concern for structural components, like the IHX and the associated piping, as they operate outside the intense radiation field. It has been found that alloy 617 exhibits higher internal oxidation and higher total corrosion rate than alloy 230.

Figure 3.2 shows the variation of yield strength and ultimate tensile strengths with temperature of as-received Alloys 617 and 230 [15]. It is evident from Fig. 3.2a, the yield strength of Alloy 617 drops from room temperature up to about 400 °C; remains roughly constant between 400-800 °C; beyond which it drops again. It can be inferred that the strength of Alloy 230 is greater than the strength of Alloy 617.
Figure 3.2   Mechanical properties of as-received alloys 617 and 230 (a) Yield Stress vs. Temperature and (b) UTS vs. Temperature
Effect of Helium Environment on Creep Properties

The effect of impure helium environment at high temperatures on the corrosion resistance of the materials has been the subject of numerous investigations [16, 17]. Helium, in all likelihood, will be the primary coolant for VHTRs. Alloy 617 is known to corrode actively above 920 °C in impure helium environment [13]. Therefore, it is important and worthwhile to understand the influence of helium environment at high temperatures on the corrosion and mechanical properties of alloy 617.

Helium coolant in VHTR is not pure and inevitably contains small amounts (of the order of ppm) of contaminants, such as H₂, H₂O, CO₂, CO, CH₄, and O₂. The impurities in helium mainly result from the degassing of adsorbed species from the massive graphite structures, from air ingestion or in-leakage. Shankar et al. [17] investigated the uniaxial creep behavior of Alloy 617 in helium environments with controlled amounts of impurities. More precisely, the creep response of alloy 617 was evaluated at 843 and 950 °C at 1 atm in three different helium environments: pure helium (He), helium with 675 vppm (parts per million by volume) methane (He + CH₄), helium with 500 vppm oxygen (He + O₂). The nominal composition of pure helium used was He-99.999%, O₂ <1 ppm, H₂O <2 ppm, and total hydrocarbons <1 ppm. Figure 3.3 shows the creep behavior of alloy 617 in a pure helium environment at 843 and 950 °C at different applied stresses [17]. Figure 3.4 compares the creep response of Alloy 617 in the three aforementioned helium environments at 843 and 950 °C. It is evident from the figure that the rupture life is the longest in He + CH₄ environment and shortest in He + O₂ environment [17]. The reduction in rupture life in He + O₂ environment is more pronounced at 950 °C compared to 843 °C. The discussion on possible metallurgical reasoning behind this behavior is beyond the scope of this thesis. However, what is
important is the fact that helium environment does influence the high-temperature properties of alloy 617.

Figure 3.3  Creep strain vs. time for Alloy 617 in pure helium environment (a) at 843 °C and (b) at 950 °C
Figure 3.4  Effect of methane and oxygen impurities in helium on the creep rupture response of Alloy 617 (a) at 843 °C and 68.95 MPa and (b) at 950 °C and 27.6 MPa.

Thermal Aging / Thermal Stability

Burlet et al. [15, 16] have performed thermal aging tests on Alloys 617 and 230 to evaluate its effect on the microstructure and the mechanical properties of the alloys. Tests have been performed for exposure times up to 5000 h and at temperatures of 700, 750, 850, and 950 °C. Preliminary results indicate an increase of strength and a
decrease of ductility for Alloy 617 while Alloy 230 suffers a slight decrease of strength and ductility. Figure 3.5a compares the UTS of unaged, 1000 h aged, and 5000 h aged Alloy 230. It is clear that the UTS of Alloy 230 was not affected by aging up to 5,000 h. Fig. 3.5b shows that the ductility of 5000 h aged Alloy 230 at 750 °C is much lower than in the unaged condition. Furthermore, it was observed that the loss of ductility is maximum at 700-750 °C for both the alloys and the loss is more pronounced for Alloy 617. These results indicate that the microstructure of Alloy 230 is still evolving after 5,000 h of aging and that there is a need for longer testing periods.

Low cycle fatigue tests by Cabet et al. [13] indicated that Alloy 230 has better fatigue performance than Alloy 617 due to its smaller grain size. In a slightly oxidizing atmosphere and for exposure periods of 813 hrs, Alloy 230 showed slightly better corrosion resistance than Alloy 617. Longer exposure periods (> 5000 h), however, are needed to determine if Alloy 230 has a better oxidation resistance than Alloy 617.
3.4.2 Alloy 230 (UNS N06230)

Alloy 230 is a very recently developed alloy and, like Alloy 617, is a leading candidate material for high-temperature applications. Alloy 230 is a solid-solution strengthened nickel-chromium-tungsten-molybdenum alloy having excellent high-temperature strength, outstanding resistance to oxidizing environments, and excellent long-term thermal stability. Tungsten and molybdenum in combination with the alloy's high carbon content impart strength, while chromium is responsible for resistance to high temperature corrosion. In the recent years, tungsten tends to replace molybdenum as an additive element for high-temperature reinforcement [13].

Allowable design stresses for Alloy 230 products are found in Table 1B of Section II, Part D of the ASME Boiler and Pressure Vessel Code. Alloy 230 is approved for ASME Section VIII, Division I construction to 1650 °F. Alloy 230 possesses good hot workability and weldability. It has a slightly lower thermal expansion coefficient than alloy 617 and a
similar thermal conductivity. Preliminary studies have shown that it has better thermal stability and oxidation resistance than Alloy 617. However, Alloy 230 is not yet qualified for VHTR application and its hot corrosion resistance in helium environments encountered in GCRs has not yet been investigated.

Figure 3.6 shows the variation of tensile properties of Alloy 230 with temperature [18]. Burlet et al. [15] compared the tensile properties of Alloys 617 and 230, as shown in Figure 3.2. It is clear that Alloy 230 has superior tensile properties than Alloy 617 up to about 750-800 °C, beyond which the difference is negligible. However, the ductility of Alloy 230 is less than Alloy 617 and the difference is not significant at higher temperatures.

Figure 3.6  Tensile properties of hot-rolled and 1232 °C solution annealed plate

Preliminary data on the low-cycle fatigue behavior of Alloy 230 in air for the temperature range of 760-982 °C indicated a better fatigue response than Alloy 617.
Cabet et al. [13] have investigated the oxidation resistance of three high-temperature alloys, namely, Alloy 617, Alloy 230, and Alloy HX exposed for 813 hours in impure helium at 950 °C. Experiments were conducted with a helium flow rate of 15 L/h and a helium composition of \(H_2 = 189 \pm 6 \text{ ppm}, \ CH_4 = 20.1 \pm 0.5 \text{ ppm}, \ CO = 50.6 \pm 1.4 \text{ ppm}, \ H_2O \sim 2.1 \text{ ppm}, \ N_2 < 8 \text{ ppm}, \ O_2 < 0.1 \text{ ppm}\). Their analysis revealed that Hastelloy X has the best oxidation resistance followed by Alloy 230 and Alloy 617. However, the time of exposure is too short to arrive at a conclusion on the corrosion resistance ability of the alloys.

Generally speaking, the high temperature resistance of Ni based Cr rich alloys is related to the formation of a dense and adherent chromia surface scale that protects from further rapid corrosion [19]. Studies by Rouillard et al. [19] on the high temperature corrosion properties of Haynes 230 in an impure helium environment revealed that the protective surface oxide scale is destroyed at temperatures greater than a critical temperature (typically 1173 K). This destructive reaction impairs the corrosion resistance ability of the alloy. Their helium composition for total pressure of 101,325 Pa is \(H_2 = 19.3 \pm 0.4 \text{ Pa}, \ CO = 4.9 \pm 0.1 \text{ Pa}, \ CH_4 = 1.9 \pm 0.04 \text{ Pa}, \ H_2O = 0.16 \pm 0.08 \text{ Pa}\). Destruction of oxide surface beyond a certain temperature is also true for other chromium containing alloys like Alloy 617, and Hastelloy X. Therefore, more studies are required to assess the suitability of these alloys for VHTR applications.

### 3.4.3 Alloy 800H and 800HT (UNS N08810/N08811)

Alloy 800H/HT is an iron-base solid-solution strengthened alloy with large additions of nickel (30-35 wt.%) and chromium (19-23 wt.%). Alloy 800HT has nearly identical chemical composition limits as Alloy 800H with the exception of carbon, aluminum and
titanium contents. The carbon content in Alloy 800HT is restricted to 0.06-0.1% as opposed to 0.05-0.1% in Alloy 800H while the Al plus Ti content of Alloy 800HT is restricted to 0.85-1.2%. While nickel and chromium ensure good oxidation and carburization resistance, carbon imparts high temperature strength and resistance to creep and rupture. This alloy must be solution annealed at high temperatures (about 1095 °C) so that the carbon is in the condition to make its optimum contribution to high temperature properties. Chromium promotes the formation of a protective surface oxide and nickel enhances the protection, especially during cyclic exposure to high temperatures. Generally, addition of Fe to Ni-Cr alloys reduces internal oxidation.

Alloy 800H is currently approved under ASME B&PVC Subsection NH for use up to 760 °C. For the pebble bed reactor design, Alloy 800H is a candidate material for control rod sheath materials. Allowable design stresses for alloy 800H/HT products are found in Table 1B of Section II, Part D of the ASME B&PVC. Alloy 800H is approved for ASME Section VIII, Division I construction to 1800 °F, while Alloy 800HT is approved to 1650 °F.

3.4.4 Hastelloy X (UNS N06002)

Hastelloy X is a nickel-chromium-iron-molybdenum alloy with good oxidation resistance at temperatures up to 1200 °C and moderately good strength properties at temperatures up to 870 °C. Chromium, molybdenum, and tungsten provide solid-solution strengthening. Japanese HTTR program is evaluating Hastelloy X and Hastelloy XR for their high-temperature metallic components [20]. Hastelloy XR, primarily developed to have sufficient compatibility with impure helium environment at high temperatures, is an improved version of Hastelloy X and was developed by optimizing the contents of certain elements. For example, optimizing manganese and silicon contents in Hastelloy X helps
in forming a stable and adherent oxide film on the surface of the base metal, while lowering aluminum and titanium contents helps in suppressing the internal oxidation.

Allowable design stresses for alloy HX products are found in Table 1B of Section II, Part D of the ASME B&PVC. Alloy HX is approved for ASME Section VIII, Division I construction to 1650 °F. As discussed in Section 3.4.2, Cabet et al. [13] investigated the corrosion response of Alloys 617, 230, and Hastelloy X coupons exposed to an impure helium environment at 950 °C for 813 hrs. Preliminary results showed that Hastelloy X has superior corrosion resistance compared to Alloys 230 and 617. However, longer tests are required to arrive at a definite conclusion on the corrosion resistance ability of these alloys.

3.5 Comparison of Other Physical Properties

Figure 3.7 compares the creep rupture behavior of Alloys 617, 230, HX, and 800H in an air environment at 982 °C [21]. It is clear that alloys 617 and 230 have the best creep rupture behavior followed by alloy HX and then alloy 800H. Figure 3.8 compares the allowable design stress values at 900 °C for various high temperature materials. Table 3.3 compares the thermal conductivity and mean coefficient of thermal expansion for different candidate materials. Clearly, alloys 617 and 230 have a low mean thermal expansion coefficient (lower thermal stresses in service) and better thermal conductivity among the candidate materials.
Figure 3.7 Creep rupture strength of Alloys 800H, HX, 230, and 617 [21]

Figure 3.8 Comparison of allowable design stress values

56
Table 3.5  Thermal conductivity and mean thermal expansion coefficient for various candidate materials at 900 °C

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Thermal Conductivity (Wm⁻¹K⁻¹)</th>
<th>Thermal Expansion Coefficient (µm·m⁻¹K⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>617</td>
<td>27.1</td>
<td>15.8</td>
</tr>
<tr>
<td>230</td>
<td>26.4</td>
<td>15.7</td>
</tr>
<tr>
<td>HX</td>
<td>26.3</td>
<td>16.3</td>
</tr>
<tr>
<td>HR160</td>
<td>26.9</td>
<td>17.1</td>
</tr>
<tr>
<td>800H</td>
<td>27.1</td>
<td>18.5</td>
</tr>
</tbody>
</table>

3.6  Materials for High-Temperature Helium Test Facility and PCHEs

Based on the materials investigation discussed above and from the considerations of economics and availability, high-temperature materials were identified for the helium test facility piping and the printed circuit heat exchangers. The current study indicated that either alloy 617 or alloy 230 is the most suitable material for the high-temperature and high pressure (up to 7.0 MPa) applications. At the time when this study was carried out, alloys 617 and 230 were available only in plate form (and not in tube or pipe form), which precluded their usage for the test facility piping. Although, alloys 617 and 230 have comparable strengths and mechanical properties at high temperatures, alloy 617 was preferred over alloy 230 for the PCHEs for the following reasons. Alloy 617 is well studied and researched in relation to Alloy 230 for high-temperature applications. The etchability of alloy 617 and the diffusion bonding procedures have been confirmed during this study. For the test facility piping needs, Alloy 800HT was selected based on economics and availability constraints.
3.7 Conclusions

Following are some salient conclusions of the material research:

a) Based on creep resistance above 850 °C, Alloys 617 and 230 are the leading candidate alloys for the NGNP project. Alloy 617 is definitely the most creep resistant among all the high-temperature alloys; however, in the presence of impure helium coolant, it is found to actively corrode above 920 °C. Issues pertaining to its longtime usage under the reactor environmental and loading conditions still need to be addressed.

b) Efforts are on the way to develop a controlled chemistry specification for Alloy 617 and other alloys to optimize the long-term properties at elevated temperatures and to minimize the effects of exposure to impure helium environment at those temperatures. Evolution of Hastelloy XR from Hastelloy X illustrates the importance of optimizing the contents of various elements to improve the properties.

c) Issues related to joining and welding need to be addressed. Mechanical testing and aging studies on the weldments of the potential candidate materials are still under investigation.

d) The corrosion resistance of Alloy 230 is unknown in the reactor specific environment. Depending on the impurity levels in the helium and the temperature, many of these high-temperature alloys can undergo oxidation, carburization, or decarburization. It is generally agreed that the concentration of H₂O and CO is important as they control the oxygen partial pressure and carbon activity, respectively.
e) The effect of radiation environment on the mechanical and the physical properties of the potential candidate materials need to be studied.

f) Current studies for exposure times up to 5000 h indicate that alloy 617 exhibits higher internal oxidation and higher total corrosion rate than alloy 230.

g) For the current design, Alloy 617 was selected as the plate material for the heat exchangers and Alloy 800HT for the PCHE header material, test facility piping, pipe fittings, and flanges.
CHAPTER 4

DESIGN OF HIGH-TEMPERATURE HELIUM TEST FACILITY

4.1 Introduction

This chapter introduces the design aspects of the high-temperature helium test facility that is being constructed at The Ohio State University. The helium test facility was envisaged to experimentally investigate the thermal hydraulic characteristics of a compact diffusion-bonded heat exchanger having potential applications in VHTRs. Some of the salient loop components, including the heaters, cooler, gas boosters, and flow meters are discussed. The initial test facility design catered to the operating temperatures and pressures up to a maximum of 950 °C and 7 MPa, respectively. However, from the consideration of materials availability and economics, the operating temperatures and pressures were scaled down and the test facility was redesigned to facilitate operation at temperatures and pressures up to 900 °C and 3 MPa, respectively.

4.2 Layout of the Test Facility

Figure 4.1 shows the layout of the high-temperature helium test facility that is being constructed at The Ohio State University. Two gas boosters in parallel provide the necessary head for circulating helium gas in the loop. Originally, a gas compressor was chosen in the design, but was not adopted due to its higher cost. A 5-gallon stainless steel tank at the exit of the gas boosters smoothen out any pressure fluctuations that may be induced by the reciprocating action of the boosters. After exiting the gas
boosters, helium gas is heated by a pre-heater with a maximum capacity of about 23 kW. The pre-heater is actually a combination of three radiant type heaters, each having a maximum capacity of 7.6 kW, and arranged in a 3-phase delta configuration. Helium gas leaving the pre-heater with a desired temperature is then forwarded to the cold side of the PCHEs. The loop is designed such that the fluid exiting the cold side of the heat exchangers enters the hot side of the PCHEs through a main heater that heats the helium to around 900 °C when operated at its maximum capacity of 23 kW. The main heater is also a combination of three radiant type heaters and is electrically configured in the same way as the pre-heater. After transferring energy to the cold side, the helium gas enters a cooler, where it transfers heat to the process chilled water and gets cooled down to the inlet temperature of the gas boosters, i.e., around 120 °C. The bypasses in the loop serve to decouple the hot and cold sides of the PCHE and help in realizing different flow rates on the hot and the cold sides. The loop is equipped with sensors for measuring the pressure and differential pressure on both the hot and cold sides of the PCHEs. K-type Alloy 800HT sheathed thermocouples are used for measuring the temperature at various locations in the loop. Three venturi type flow meters measure the volumetric flow rates of helium gas flowing through the loop. Additionally, two high-temperature flow sensors designed by Delta M Corporation will be installed in the loop for prototype design testing and cross benchmark of the flow measurements. A flow meter installed on the process chilled water side of the cooler allows monitoring the flow rate of the process chilled water. Along with the information of the inlet and exit temperatures of the chilled water, this allows us to calculate the rate of the energy being removed by the chilled water.
The present design allows us to perform experiments up to a maximum temperature of 900 °C and a wide range of pressures (0.5 to 3.0 MPa) and flow rates (10 to 80 kg/h).

Figure 4.1  Layout of the high-temperature helium test facility

4.3 Design of the High-Temperature Helium Test Facility

In what follows, the design aspects of the helium test facility are presented. This includes the piping size estimation, the material selection for the piping and the printed circuit heat exchangers, and the estimation of piping pressure design thickness for the maximum operating conditions. The current test facility design facilitates operation at temperatures and pressures up to 900 °C and 3 MPa, respectively. However, the initial
design was conceived for operating temperatures and pressures up to 950 °C and 7 MPa, respectively. This design had to be modified due to budget constraints and non-availability of the material in the required pipe form. The current design is based on ASME B31.3 process piping code and ASME Section II, Part D for material properties and allowable design stresses. Constraints imposed by ASME B&PVC Section III have not been employed in the design and hence the design is not intended for direct nuclear applications.

4.3.1 Operating Conditions

To summarize, the high-temperature helium test facility is designed with the following specifications:

a) Maximum operating temperature: 900 °C
b) Maximum operating pressure: 3 MPa
c) Working fluid: Helium gas
d) Range of mass flow rates on both the hot and cold sides: 10-80 kg/h

For the reasons discussed in Section 4.3.2, the maximum operating temperature and pressure for the test facility was scaled down to 900 °C and 3 MPa, respectively. For the current experiments, helium was selected as the working fluid as the reference NGNP concept utilizes helium gas as the primary coolant. In addition, till date, the literature shows that the thermal-hydraulic performance of PCHEs has not been investigated with helium as the working fluid and at operating conditions typical of VHTR. With appropriate modifications, the loop can also be employed for testing PCHEs with other working fluids, such as N₂ and He mixture, water, etc. The range of flow rates selected were
determined based on the current PCHE design and allows it to be operated in laminar and laminar-to-turbulent transition regimes.

4.3.2 Line (Piping) Size Estimation

Piping in the test loop conveys the working fluid between various components. Line size was estimated based on the velocity limitation of 25 m/s in the line. This economic design velocity of 25 m/s is dictated by pipe material and erosion and corrosion considerations [22]. The pipe material was not determined when the line size was estimated. Hence, for the purposes of line size estimation, austenitic stainless steel was assumed as the pipe material. The range of helium mass flow rates for the proposed experiments range from 10-80 kg/h. From the knowledge of the mass flow rates and the design velocity limitation of the working fluid in the line, the line size can be estimated. Table 4.1 summarizes both the initial and scaled down operating conditions and other relevant data utilized for approximating the line size. The line size is estimated as,

\[ \dot{m}_{\text{max}} = \rho_{He} A V_{He} = \rho_{He} V_{He} \frac{\pi}{4} d^2 \]  

(4.1)

where \( \rho_{He} \), \( V_{He} \), \( A \), and \( d \) are the density of helium gas, the economic design velocity of helium in the pipe, cross-sectional area of the pipe, and the inner diameter of the pipe, respectively. Considering the maximum mass flow rate of 80 kg/h and helium properties at 7.0 MPa and 950 °C (initial design operating parameters), the inner diameter of the pipe is given by,

\[ d = \sqrt{\frac{4 \dot{m}_{\text{max}}}{\pi \rho_{He} V_{He}}} = 20.3 \ mm \]  

(4.2)
Table 4.1  Design parameters for line size estimation

<table>
<thead>
<tr>
<th>Working fluid</th>
<th>Operating temperature (°C)</th>
<th>Operating pressure (MPa)</th>
<th>Mass flow rates kg/h</th>
<th>Density $\rho_{He}$ (kg/m³)</th>
<th>Economic design velocity $V_{He}$ (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial</td>
<td>Helium</td>
<td>950</td>
<td>7.0</td>
<td>5 80</td>
<td>2.74 25</td>
</tr>
<tr>
<td>Scaled</td>
<td>Helium</td>
<td>900</td>
<td>3.0</td>
<td>5 80</td>
<td>1.23 25</td>
</tr>
</tbody>
</table>

Material Selection for Piping

Material selection for piping was a critical issue in the design of the high-temperature helium test facility. The choice of material for the piping was basically governed by the operating temperatures and pressures, availability, and economics. Based on the high-temperature materials investigation carried out as a part of this research, five high-temperature materials were selected as potential piping materials. A systematic pressure design analysis of the piping for the maximum operating temperatures and pressures was carried out and based on availability and economics, Alloy 800HT was selected as the piping material.

4.3.3 Piping Pressure Design Thickness Estimation

4.3.3.1 Basic Allowable Stresses

ASME B31.3 covers process piping and ASME B31.7 (now moved to B&PVC Section III) deals with nuclear piping [23]. For the purposes of the present design, ASME B31.3 was used for piping. Design conditions in ASME B31.3 are specifically intended for pressure design. The design pressure and temperature are the most severe coincident conditions, i.e., conditions that result in the greatest pipe wall thickness.
For the purposes of estimating the pressure design thickness for the piping, basic allowable stresses from ASME B31.3 were used. For materials like Alloys 617 and HR160, which are not covered by ASME B31.3, maximum allowable stresses for estimating the pressure design thickness were taken from ASME B&PVC Section II, Part D. The allowable stress for a piping system or a piping component material is based on a function of the yield or tensile strength of the material at cold to moderate temperatures, or is based on creep rates or stress for rupture in elevated temperature service. The basic allowable stress values are tabulated in Appendix A, Table A-1 of the B31.3 Code for various materials. The designer is permitted to linearly interpolate between temperatures for which allowable stresses are listed. Also, use of materials above the maximum temperature for which allowable stresses are provided is permitted as long as it is not specifically prohibited.

Generally, creep range temperatures are those above 700 to 800 °F. Basic allowable stress values at temperature shall not exceed the lowest of the following [23]:

a) \( \frac{1}{3} \) of specified minimum tensile strength \( (S_T) \) at room temperature

b) \( \frac{1}{3} \) of tensile strength \( (1.1S_TR_T) \) at temperature

c) \( \frac{2}{3} \) of specified minimum yield strength \( (S_Y) \) at room temperature

d) \( \frac{2}{3} \) of yield strength at temperature (for austenitic stainless steels and certain Ni alloys, this value may be as large as 90% of yield strength at temperature)

e) 100% of the average stress for a creep rate of 0.01% per 1000 h

f) 67% of the average stress for creep rupture in 10,000 h

g) 80% of the minimum stress for creep rupture in 10,000 h
4.3.3.2 Pressure Design of Straight Pipe for Internal Pressure

The design expressions in this section are taken from ASME B31.3 Process Piping Code [23]. The minimum thickness of the pipe selected, considering manufacturers minus tolerance, must be at least equal to $t_m$, as defined below.

$$t_m = t + c$$  \hfill (4.3)

where

- $c$ = the sum of the mechanical allowances plus corrosion and erosion allowances
- $t$ = pressure design thickness
- $t_m$ = minimum required thickness including allowances
For pipe with \( t < D/6 \), the following basic equation for determining pressure thickness is provided in the Code.

\[
t = \frac{PD}{2(SE + PY)}
\]  \hspace{1cm} (4.4)

where

\( D \) = pipe outside diameter (not nominal diameter), mm

\( E \) = quality factor

\( P \) = internal design gage pressure, MPa

\( S \) = allowable stress value, MPa

\( Y \) = coefficient provided in Table 304.1.1 of the Code and here Table 4.2

The \( E \) factor is an “allowable pressure stress penalty” based on the method of manufacture of the pipe. It reflects the quality of the longitudinal weld in seam-welded pipe and will have a value ranging from 0.6 for furnace butt welded (FBW) to 1.0 for seamless pipe (SMLS) [24]. The \( Y \) factor is included to account for the non-linear reduction in allowable stress at design temperatures above 482 °C (900 °F).

The outside diameter of pipe is independent of wall thickness. Equation (4.4) is based on outside diameter of pipe which makes the calculation of wall thickness straightforward. Equation (4.4) above is called the Boardman equation and is an
empirical approximation of the more accurate and complex Lame equation. Boardman equation provides a good match to the more accurate and complex Lame equation for a wide range of diameter to thickness ratios and becomes increasingly conservative for lower $D/t$ ratios (thicker pipes).

Table 4.2 Values of Coefficient $Y$ for $t < D/6$
(B31.3, Table 304.1.1)

<table>
<thead>
<tr>
<th>Temperature, °C (°F)</th>
<th>Materials</th>
<th>$\leq 482$ ($\leq 900$)</th>
<th>510 (950)</th>
<th>538 (1000)</th>
<th>566 (1050)</th>
<th>593 (1100)</th>
<th>$\geq 621$ ($\geq 1150$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ferritic steels</td>
<td>0.4</td>
<td>0.5</td>
<td>0.7</td>
<td>0.7</td>
<td>0.7</td>
<td>0.7</td>
<td></td>
</tr>
<tr>
<td>Austenitic steels</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
<td>0.5</td>
<td>0.7</td>
<td></td>
</tr>
<tr>
<td>Other ductile metals</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
<td></td>
</tr>
<tr>
<td>Cast iron</td>
<td>0.0</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td></td>
</tr>
</tbody>
</table>

For the thicker wall pipes, ASME B31.3 provides the following equation for the calculation of the $Y$ factor.

$$Y = \frac{d + 2c}{D + d + 2c}$$ (4.5)

The factor $Y$ depends on temperature. At elevated temperatures, when creep effects become significant, creep leads to a more even distribution of stress across the
pipe wall thickness. Thus, the factor $Y$ increases, leading to a decrease in the calculated, required wall thickness (for a constant allowable stress).

ASME B31.3 provides three other equations.

$$t = \frac{PD}{2SE} \quad (4.6)$$

Equation (4.6) is the simple Barlow equation, which is based on the outside diameter and is always conservative. It may be used since it is always more conservative than the Boardman equation, which is based on a smaller diameter (except when $Y = 0$).

$$t = \frac{D}{2} \left(1 - \frac{SE - P}{\sqrt{SE + P}}\right) \quad (4.7)$$

Equation (4.7) is the Lame equation rearranged to calculate thickness.

The following optional equation remains in ASME B31.3

$$t = \frac{P(d + 2c)}{2[SE - P(1 - Y)]} \quad (4.8)$$

where $d$ is inside diameter

4.3.3.3 Initial Design

As mentioned before, the high-temperature helium test facility was initially designed to facilitate operation at temperatures and pressures up to a maximum of 950 °C and 7 MPa, respectively. In what follows, pressure design thickness (wall thickness minus the corrosion and erosion allowances) for the high temperature materials listed in Section
4.3.3.2 was estimated for operating temperatures up to 950 °C and at maximum operating pressure of 7 MPa. Boardman expression, namely equation (4.4), was used for estimating the pressure design thickness required for a seamless 1 inch nominal pipe size (N.P.S).

Figure 4.3 shows the variation of pressure design thickness with temperature for different high-temperature materials at an internal design gage pressure of 7 MPa. From Fig. 4.2, it is evident that, among the materials, only Alloys 617, 230, and 800H are approved by ASME for temperatures up to 950 °C. For the high-temperature materials considered, the maximum commercially available wall thickness for a 1-inch N.P.S is 6.35 mm (Schedule 160). It is obvious that the pressure design thickness corresponding to 7 MPa and 950 °C far exceeds the commercially available wall thickness of 6.35 mm. In fact, at 7 MPa and 950 °C, even the best high-temperature material (Alloy 617 or 230) has a thickness requirement that is approximately twice the maximum commercially available pipe wall thickness. Pipes with such heavy walls can only be realized by extruding them out of a solid bar. Considering the budget constraints, extruding the pipe out of a solid bar was far from possible. Besides, it was more important to analyze the performance behavior of PCHEs at higher temperatures than at higher pressures. The design operating pressure was then scaled down from 7 MPa to lower pressures. Furthermore, non-availability of high-temperature materials like Alloys 617, 230, and HX in pipe form and limitations on ASME allowable stress values and maximum permitted temperatures for the other materials caused the maximum operating design temperature to be scaled down from 950 °C to 900 °C. From the consideration of economics and availability, Alloy 800HT was selected as the material for piping. In what follows, the pressure design thickness requirement for Alloy 800HT at different pressures and
temperatures up to 900 °C was estimated and the maximum operating conditions determined. The allowable design stresses for evaluating the wall thickness are from Appendix B Table B.1 of the ASME B31.3 Process Piping Code.

![Figure 4.3](image.png)

**Figure 4.3** Pressure design thickness vs. temperature

### 4.3.3.4 Current Piping Design

Alloy 800HT was commercially available in the required form, i.e., seamless pipe, and size, i.e., 1 in. N.P.S, Schedule 160. The pressure design thickness was then evaluated for Alloy 800H at different pressures and 900 °C to determine the design operating pressure. Figure 4.4 provides a rationale for designing the test facility for a maximum working pressure and temperature of 3 MPa and 900 °C, respectively. Noting the fact that the maximum wall thickness for a commercially available 1 inch, Sch.160 N.P.S. Alloy 800HT pipe is 6.35 mm, it can be observed from Figure 4.4 that the required pressure design thickness corresponding to about 3 MPa and 900 °C, is close
to 6.35 mm. Figure 4.5 compares the pressure design thickness for a 1 inch N.P.S Alloy 800HT pipe using three different expressions from ASME B31.3. The pressure design thickness is estimated at an internal design gage pressure of 3 MPa. It is clear that among all the three expressions, Barlow’s expression, as expected, results in a more conservative estimate, while Lame’s and Boardman expression agree with each other.

Figure 4.4  Pressure design thickness vs. temperature at different pressures
4.4 Principal Loop Components

In the following subsections, the design specifications of some of the principal components of the high-temperature helium test facility are discussed. High operating temperatures precluded the use of cost-effective off the shelf loop components. In addition, limited available budget restricted the design of some of the loop components with exotic materials. Operating within the available budget and at the same time ensuring the material integrity of the loop components for use in high temperatures was a challenge. The design process became an iterative process in that it involved repeatedly modifying the design to fit the availability and budget.

4.4.1 Electric Heater and Power Controller

Kanthal’s FIBROTHAL standard RAC tube modules were employed as heating source for heating the helium gas to temperatures of interest. The heating system
comprises of a pre-heater and a main heater. The pre-heater and the main heater are each a combination of three heaters wired in a 3-phase delta configuration with each individual heater having a maximum heating capacity of 7.6 kW. In summary, a total of six heaters with a total maximum heating capacity of about 46 kW act as the heating source to deliver helium to the PCHEs at temperatures of interest. Two phase angle type SCR (Silicon Controlled Rectifier) power control units, one for the pre-heater and other for the main heater, control the amount of power input to the heaters. Furthermore, two Honeywell UDC 2500 temperature controllers with a thermocouple input and a 4-20 mA output monitor and control the fluid temperature by providing continuous feedback to the SCR controllers and help in realizing the desired outlet temperatures. Six microprocessor-based UDC 1200 limit controllers with a thermocouple input and a relay output that is continuously fed to the power controller provide necessary safety by preventing overheating of the heating elements. Table 4.3 lists the general design specifications of the pre-heater and the main heater.

<table>
<thead>
<tr>
<th>Specification</th>
<th>Pre-Heater</th>
<th>Main Heater</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nominal inlet temperature (°C)</td>
<td>100</td>
<td>650</td>
</tr>
<tr>
<td>Maximum outlet temperature (°C)</td>
<td>300</td>
<td>850</td>
</tr>
<tr>
<td>Maximum mass flow rate (kg/h)</td>
<td>80</td>
<td>80</td>
</tr>
<tr>
<td>Working Fluid</td>
<td>Helium Gas</td>
<td>Helium Gas</td>
</tr>
<tr>
<td>Power (kW)</td>
<td>23</td>
<td>23</td>
</tr>
</tbody>
</table>
Figure 4.6 shows a Kanthal FIBROTHAL RAC tube heater with the embedded heating element and the ceramic fibre insulation. Table 4.4 lists the electrical specifications of the FIBROTHAL RAC tube heater. Each FIBROTHAL heater consists of vacuum-formed ceramic fibre components with radiating heating element embedded into it. The ceramic fibre used in these heating modules is labeled as F-17 and Table 4.5 lists the technical data for the same. The embedded heating element is a Kanthal Grade A-1 heating element with a nominal chemical composition (% by weight) of 22% chromium, 5.8% aluminum, and balance iron. The heating elements are designed for a maximum element temperature of 1300 °C.

![Figure 4.6 Kanthal RAC Fibrothal tube heater with embedded heating element](image)

<table>
<thead>
<tr>
<th>Type designation</th>
<th>Voltage (V) Power (W) at 60 A</th>
<th>Voltage (V) Power (W) at 72 A</th>
<th>Voltage (V) Power (W) at 85 A</th>
<th>Resistance R20 (Ohm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RAC 70/500</td>
<td>63.1</td>
<td>75.8</td>
<td>89.5</td>
<td>1.008</td>
</tr>
</tbody>
</table>
Table 4.5  Technical data of ceramic fibre F-17 module

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Ceramic Fibre F-17</strong></td>
<td></td>
</tr>
<tr>
<td>Maximum continuous duty temperature (°C)</td>
<td>1300</td>
</tr>
<tr>
<td>Guide analysis (%):</td>
<td></td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>50</td>
</tr>
<tr>
<td>SiO₂</td>
<td>50</td>
</tr>
<tr>
<td>Thermal conductivity (W/mK)</td>
<td></td>
</tr>
<tr>
<td>200 °C</td>
<td>0.07</td>
</tr>
<tr>
<td>400 °C</td>
<td>0.10</td>
</tr>
<tr>
<td>600 °C</td>
<td>0.14</td>
</tr>
<tr>
<td>800 °C</td>
<td>0.21</td>
</tr>
<tr>
<td>1000 °C</td>
<td>0.29</td>
</tr>
<tr>
<td>1200 °C</td>
<td>0.41</td>
</tr>
<tr>
<td>1300 °C</td>
<td>0.49</td>
</tr>
</tbody>
</table>

### 4.4.2 Cooler

The cooler is a tube-in-tube heat exchanger and uses process chilled water (PCW) as a cooling medium to cool the hot helium gas exiting the PCHEs to the inlet temperature of the gas boosters. Figure 4.7 shows the cooler that will be used for cooling the helium gas. The specifications of the cooler are as shown in Table 4.6. A mass flow meter and two thermocouples on the PCW line of the cooler enables measuring the mass flow rate and inlet and exit temperatures of the process chilled water.
Figure 4.7  Cooler for cooling helium gas

Table 4.6  Cooler design specifications

<table>
<thead>
<tr>
<th></th>
<th>Inner Tube</th>
<th>Outer Tube</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outside Diameter (inch)</td>
<td>½</td>
<td>1</td>
</tr>
<tr>
<td>Fluid Type</td>
<td>Helium</td>
<td>Water (PCW)</td>
</tr>
<tr>
<td>Inlet Temperature (°C)</td>
<td>450</td>
<td>20</td>
</tr>
<tr>
<td>Outlet Temperature (°C)</td>
<td>100</td>
<td>27</td>
</tr>
<tr>
<td>Mass Flow (kg/s)</td>
<td>0.022</td>
<td>1.382</td>
</tr>
<tr>
<td>Volumetric Flow (Lpm)</td>
<td>389.9</td>
<td>83.28</td>
</tr>
<tr>
<td>Pressure Drop (kPa)</td>
<td>82.26</td>
<td>93.32</td>
</tr>
<tr>
<td>Heat Transfer (kW)</td>
<td>40.4</td>
<td></td>
</tr>
<tr>
<td>Effectiveness</td>
<td></td>
<td>0.81</td>
</tr>
</tbody>
</table>
4.4.3 Vacuum Pump

Before charging the loop with helium, the loop is sufficiently vacuumeed with the help of a DuraVane\textsuperscript{HV} RVR002H high vacuum pump. This procedure is carried out to minimize the amount of air in the working fluid. A final vacuum pressure of $7.5 \times 10^{-2}$ Torr (9.99e-05 bar) can be realized with this pump.

4.4.4 Gas Boosters

The purpose of the gas boosters is to provide the driving head for circulating helium gas in the loop. Two gas boosters in parallel provide the necessary head and flow rate for circulating helium gas in the loop. Initially, a compressor was used in the design. However, cost considerations encountered in the design dictated the choice of gas booster over the compressor. A gas booster differs from the compressor in that it is air-driven and requires no electrical motive force. Haskel's 8AGD-2.8 model gas booster, shown in Fig. 4.8, was selected for the current application. This is a single stage, double acting, high flow, air driven, and reciprocating piston type non-lube-oil free gas compressor. Table 4.7 lists the specifications of 8AGD-2.8 gas booster.

A gas booster essentially consists of an air drive section (drive cylinder) and a gas barrel section (boost cylinder) isolated from each other by appropriate seals. The piston in the drive cylinder is attached to the piston in the boost cylinder. As the drive piston reciprocates, it compresses the gas in the boost cylinder. The maximum pressure boost is equal to the drive piston area divided by the boost piston area multiplied by the pressure feeding the drive cylinder. In other words,

$$P_o = \frac{A}{A_b} P_a + P_s$$  \hspace{1cm} (4.9)
where $A_d$, $A_b$, $P_o$, $P_a$, and $P_s$ are the drive piston area, boost piston area, gas outlet pressure, drive pressure, and gas supply pressure, respectively. For the gas booster 8AGD-2.8, the approximate area ratio of the air drive piston area to gas piston area is 2.8.

Double acting, single stage, single air head 8AGD–2.8

Net weight 55 Kg
Boxed weight 60 Kg
Box dimensions 100 x 60 x 40 cm

Inlet Gas Port: 1/2" NPT (2 ea.)
Outlet Gas Port: 1/2" NPT (2 ea.)

Figure 4.8 Haskel 8AGD-2.8 gas booster
Table 4.7  Haskel 8AGD-2.8 gas booster specifications

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum Rated Gas Supply (psig)</td>
<td>800</td>
</tr>
<tr>
<td>Maximum Rated Gas Outlet (psig)</td>
<td>800</td>
</tr>
<tr>
<td>Static Outlet (Stall) Pressure Formula</td>
<td>$2.8P_a + P_s$</td>
</tr>
<tr>
<td>Piston Displacement</td>
<td>125 in$^3$ per Cycle</td>
</tr>
<tr>
<td>Minimum Inlet Gas Pressure (psi)</td>
<td>100</td>
</tr>
<tr>
<td>Max. Outlet Gas Pressure (psi)</td>
<td>800</td>
</tr>
<tr>
<td>Max. Compression Ratio</td>
<td>25:1</td>
</tr>
</tbody>
</table>

Figure 4.9  Performance curve for 8AGD-2.8 gas booster
The compressed air plant in Scott Laboratory at The Ohio State University is rated for over 430 SCFM at 100 psig. Based on the performance curve of the 8AGD-2.8 gas booster, 40 psig drive air at 20 SCFM (Standard Cubic Feet per Minute) would result in a helium gas flow rate of 108.6 SCFM per booster. Hence, two gas boosters in parallel help to realize a maximum flow rate of around 216 SCFM which is equivalent to a flow rate of 62 kg/h. The leakage rate from the gas booster is 0.1 SCFH (Standard Cubic Feet per Hour).

4.5 Working Fluid

Helium gas is employed as the working fluid for performance testing of PCHEs. Research grade helium (99.9999% pure) is selected for the experiments. The certificate of analysis is attached in Appendix C.

4.6 Instrumentation

4.6.1 Flow Meter

Three Venturi type flow meters with NIST traceable certification and placed at appropriate locations in the test facility measure the volumetric flow rate of helium gas with an accuracy of $\pm 0.2\%$ of reading. The flow meter after the gas boosters and before the pre-heater measures the total volumetric flow rate while the other two enable the measurement of the bypass flow rates on the hot and cold sides of the PCHEs. Additionally, two Delta M high temperature mass flow sensors designed by Delta M Corporation will be installed in the test facility for prototype testing.

4.6.2 Temperature and Pressure Sensors

Fourteen K-type standard duty socket weld thermo wells of Alloy 800HT construction are used for the measurement of temperature. The nominal accuracy of these
thermowells are around $\pm 2.2 \, ^\circ\text{C}$ in the measurement range of 100 to 900 $^\circ\text{C}$. Honeywell ST3000 smart pressure transducers with a 4-20 mA dc output are used for measuring the gage and differential pressures. In order to measure the pressure or pressure differential in the loop at temperatures above the operating range of transducers, the pressure transducer is isolated from the pressure source by a long length of tubing.

4.6.3 Data Acquisition System

All data acquisition and process control tasks are managed by a PC executing LabView 8.5 under Windows XP. The DAQ system consists of NI compact DAQ chassis and five NI 9211 modules and one NI 9205 module. The NI 9211 is a 24-bit, 4-channel thermocouple input module while the NI 9205 is a 16-bit, 32-channel single-ended or a 16-channel differential analog input module.

4.7 Test Facility Welding Issues [25]

Welding qualifications to ASME IX are for use with the ASME B&PVC or the ASME piping codes. Validated procedures for on-site welding, postweld heat treatment (PWHT), and inspections will be required for the materials of construction. Welding involves a welding procedure specification (WPS) that has been qualified by a procedure qualification test, which is a document in a procedure qualification record (PQR). Welders are required to pass a performance qualification test in order to be qualified to perform Code welding. This is documented in the welder qualification records (WQR). Materials are categorized by P-Numbers, which are groupings of alloys for weld procedure qualification purposes; they group materials based on composition, weldability, and mechanical properties. P-Numbers are assigned in Section IX of the Boiler and Pressure Vessel Code. Qualification of a welding procedure for a particular base material qualifies that procedure for any other base material with the same P-
Number. Welding qualifications carried out under the ASME code do not require independent inspection. For ease of welder qualification, the ASME Section IX “P” classification for alloys 800HT and 617 is given below in Table 4.8.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>UNS Number</th>
<th>Specification</th>
<th>P-Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>617</td>
<td>N06617</td>
<td>SB-168 (Plate, Sheet or Strip)</td>
<td>43</td>
</tr>
<tr>
<td>800HT</td>
<td>N08811</td>
<td>SB-407 (Seamless Pipe and Tube)</td>
<td>45</td>
</tr>
</tbody>
</table>

The welding consumables (electrodes and welding rods) recommended for joining

a) Alloy 800HT to Alloy 800HT: For service temperatures over 790 °C and for applications that require the highest strength and corrosion resistance, Inconel welding electrode 117 and Inconel filler metal 617 are recommended. These welding consumables have an ASME Section IX “F” Classification of “F43”.

b) Alloy 800HT to Alloy 617: For service temperatures over 790 °C and for applications that require the highest strength and corrosion resistance, Inconel welding electrode 117 and Inconel filler metal 617 are recommended. These welding consumables have an ASME Section IX “F” Classification of “F43”. INCONEL Filler Metal 617 is used for gas-tungsten-arc and gas-metal arc welding while INCONEL Welding Electrode 117 is used for shielded metal-arc welding.
CHAPTER 5

HEAT TRANSFER AND PRESSURE DROP CORRELATIONS FOR PCHE GEOMETRY

5.1 Introduction

This chapter provides an overview of the heat transfer and pressure drop correlations that are applicable to straight channels of approximately semi-circular cross-section. Unless otherwise mentioned specifically, all correlations are for circular ducts and may be applied for noncircular duct geometries with some uncertainty. These correlations can be used to arrive at a heat transfer and a pressure drop model for the PCHE. A correlation applicable to laterally corrugated (wavy) channels of semi-circular cross section is also discussed.

5.2 Printed Circuit Heat Exchanger Geometrical Details

In what follows, some of the geometrical parameters of PCHE are given. It is assumed that the PCHE has straight channels of semi-circular cross section.

a) The cross-sectional area and perimeter of a semi-circular channel are evaluated from,

\[ A_c = \frac{1}{2}\left(\frac{\pi}{4}D^2\right) \]  
(5.1)
\[ P = \frac{\pi D}{2} + D \]  \hspace{1cm} (5.2)

where \( D \), \( A_c \), and \( P \) are the channel diameter, channel flow cross-sectional area, and channel perimeter, respectively.

b) Hydraulic diameter: The hydraulic diameter of a semi-circular channel is evaluated from,

\[
D_h = \frac{4A_c}{P} = \frac{\pi D}{2\left(\frac{\pi}{2} + 1\right)}
\]  \hspace{1cm} (5.3)

where \( D_h \) is the channel hydraulic diameter.

c) Free flow area of each side (hot or cold) can be evaluated from,

\[
A = n_c A_c
\]  \hspace{1cm} (5.4)

where \( A \) is the free flow area of either the hot or cold side and \( n_c \) is the number of channels on either side (hot or cold).

d) Heat transfer area of each side can be evaluated from,

\[
A_h = n_c L_c P = n_c L_c \left(\frac{\pi D}{2} + D\right)
\]  \hspace{1cm} (5.5)

where \( A_h \) is the heat transfer area of either the hot or cold side and \( L_c \) is the length of the channel, respectively.
5.3 Heat Transfer and Pressure Drop Correlations

The information on heat transfer modeling in PCHE is limited. Heat transfer correlations for both the laminar and turbulent flow in a straight semi-circular duct are addressed in Hesselgreaves [26]. In compact heat exchanger analysis, the heat transfer coefficient is usually expressed in terms of the Colburn $j$ factor as,

$$j = \frac{N_u}{Re Pr^{1/3}} = St Pr^{2/3} = \frac{D_h}{4L} Pr^{2/3} N$$

(5.6)

$$h = \frac{j}{Pr^{2/3} Gc_p}$$

(5.7)

where $N_u$ is the Nusselt number, $Re$ the Reynolds number, $Pr$ the Prandtl number, $G$ the mass velocity, and $c_p$ the specific heat at constant pressure. $N$ is the number of thermal units (NTU) for the side and $St$ is the Stanton number defined respectively as,

$$N = \left( T_2 - T_1 \right) / \Delta T$$

(5.8)

$$St = \frac{h}{Gc_p}$$

(5.9)

where $T_1$ and $T_2$ are the end temperatures and $\Delta T$ is the mean temperature.

5.3.1 Developing and Fully Developed Laminar Flow Correlations

Unlike turbulent duct flow (for $Pr > 0.7$ fluids), the heat transfer rate in laminar duct flow is sensitive to the thermal boundary condition. Hence, it is essential to identify the thermal boundary condition in laminar flow. Figure 5.1 shows the variation of local...
Nusselt numbers for the thermal entrance region of a semi-circular duct with dimensionless thermal length from leading edge, \( x^* \). The dimensionless thermal length \( x^* \) is defined as,

\[
x^* = \frac{x / D_h}{RePr}
\]

where \( x \) is the flow length. In fully-developed laminar flow, the Nusselt number is constant and independent of Reynolds number, but its value depends on the surface thermal condition and flow passage geometry. For fully developed laminar flow in a straight semi-circular channel with axially constant wall heat flux and circumferentially constant wall temperature, Hesselgreaves [26] recommends using,

\[
Nu = 4.089
\]

A constant \( Nu \) implies that the convective heat transfer coefficient is independent of the flow velocity and the fluid Prandtl number. The product of fanning friction factor \( f \) and Reynolds number for fully developed laminar flow in a semi-circular duct as reported in Hesselgreaves [26] is,

\[
f Re = 15.78
\]
5.3.2 Transition and Fully-Developed Turbulent Flow Correlations

It is generally accepted that the hydraulic diameter correlates $Nu$ and $f$ for fully developed turbulent flow in circular and noncircular ducts. For the transition and turbulent flow regimes ($Re > 2300$), Hesselgreaves [26] recommends using the Gnielinski correlation for the smooth, straight semi-circular channels. It is given by,

$$Nu = \frac{\left(\frac{f}{2}\right)(Re - 1000)Pr}{1 + 12.7\left(Pr^{2/3} - 1\right)\sqrt{\frac{f}{2}}} \quad \text{2300} \leq Re \leq 5 \times 10^6 \text{ and } 0.5 \leq Pr \leq 2000 \quad (5.13)$$

where $Nu$ is the Nusselt number, $Re$ the Reynolds number, $Pr$ the Prandtl number, and $f$ the fanning friction factor defined as,

$$f = A + BRe^{-1/m} \quad (5.14)$$

Figure 5.1  Local Nusselt number vs. non-dimensional thermal length
where \( A = 0.0054, B = 2.3 \times 10^{-8}, m = -2/3 \) for \( 2100 \leq \text{Re} \leq 4000 \)
\( A = 0.00128, B = 0.1143, m = 3.2154 \) for \( 4000 \leq \text{Re} \leq 10^7 \)

The Prandtl number of helium is approximately given by the expression,

\[
\text{Pr} = 0.74 T^{-0.014}
\]  

(5.15)

where \( T \) is the temperature in Kelvin. Equation (5.15) shows that Eqs. (5.13) and (5.14) are applicable for helium. Equation (5.14) is a friction factor correlation proposed by Bhatti and Shah as reported in Hesselgreaves [26] for transition and fully developed turbulent flow in circular ducts. However, fair estimates of \( f \) can be obtained for a semi-circular channel by using hydraulic diameter as the characteristic dimension. The physical reasoning behind this is that in fully turbulent flow there is sufficient mixing and thus the overall flow represented by the core velocity distribution is largely insensitive to shape. It is reported in Rohsenow et al. [27] that noncircular ducts with laminar \( f \text{Re} < 16 \) have turbulent \( f \) factors lower than those for the circular tube, whereas ducts with \( f \text{Re} > 16 \) have turbulent \( f \) factors higher than those for the circular tube. Similar trends are observed for the Nusselt numbers. From Equation (5.12), it can be inferred that semi-circular ducts have turbulent \( f \) factors lower than those for circular tube.

For fully-developed turbulent flow in the fully rough circular duct of roughness \( \varepsilon \), Hesselgreaves [26] recommends the correlation by Bhatti and Shah. It is given by,

\[
\text{Nu} = \frac{\left( \text{Re} - 1000 \right) \text{Pr} \left( f / 2 \right)}{1 + \sqrt{f / 2 \left[ 17.42 - 13.77 \text{Pr}^{0.8} \right] \text{Re}^{-0.5} - 8.48}}
\]  

(5.16)
for $0.5 < Pr < 5000$, $0.002 < \varepsilon / D_h < 0.05$, and $Re > 2300$, with

$$Pr_i = 1.01 - 0.09 Pr^{0.36} \quad \text{for} \quad 1 \leq Pr \leq 145$$

In Equation (5.16), $Re_\varepsilon$ is the roughness Reynolds number, defined as,

$$Re_\varepsilon = \frac{\varepsilon u_t}{\nu} = \frac{\varepsilon}{\nu} \sqrt{\frac{\tau_w}{\rho}} \quad (5.17)$$

where $u_t$ is the turbulent friction or shear velocity, and $\tau_w$ is the wall shear stress. The value $Re_\varepsilon < 5$ corresponds to the hydraulically smooth regime; $5 \leq Re_\varepsilon \leq 70$ corresponds to the transition from the hydraulically smooth regime to the completely rough regime; and $Re_\varepsilon > 70$ corresponds to the completely rough regime [27]. From the knowledge of Nusselt number, the convection heat transfer coefficient can be estimated as,

$$h = Nu \frac{k_{fluid}}{D_{boul}} \quad (5.18)$$

where $h$ is the heat transfer coefficient and $k_{fluid}$ is the thermal conductivity of the fluid.

For wavy channels, Hesselgreaves [26] recommends using the correlation for planar corrugated wall channels developed by Oyakawa et al. [28]

$$j = 0.125 Re^{-0.36} \quad (5.19)$$
From the $j$ factor, the heat transfer coefficient for the wavy channels can be estimated from Equation (5.7). However, the usage of this correlation for a semi-circular wavy channel is questionable as it was developed for a planar corrugated wall channel.

### 5.3.3 Friction Factor Correlations

The friction factor is a parameter of paramount importance for the design of any heat exchanger. In a PCHE, channel friction factor is essential for estimating the frictional pressure drop for a given flow rate and geometry. The total pressure drop is the sum of the contributions from the friction, the form losses, and the acceleration pressure drop. The gravitational loss is ignored as it is recoverable. The frictional pressure drop can be estimated from,

$$
\Delta p_{fri} = f \frac{4L}{D_{hyd}} \frac{1}{2} \rho v^2
$$

where $L$ is the flow length of the channel, $\rho$ is the density, and $v$ is the local fluid velocity. The entrance and exit loss contribute to the form losses in the straight channels and are given by,

$$
\Delta p_{form} = K \frac{1}{2} \rho v^2
$$

where $K$ is the form loss coefficient with a value of 0.5 for entry and 1 for exit. The friction factor $f$ can be determined from a correlation.
The friction factor is dependent on the flow regime. For fully-developed laminar flow, the friction factor is independent of wall roughness and the product of the fanning friction factor and the Reynolds number is given by Equation (5.12).

The friction factor for fully-developed turbulent flow in smooth round channels can be estimated from the Colebrook equation as,

\[
\frac{1}{\sqrt{f}} = 1.5635 \ln \left( \frac{Re}{7} \right) \quad 4 \times 10^3 \leq Re \leq 10^7
\]  

(5.22)

For turbulent flow in rough channels, Colebrook-White correlation can be used and is given by,

\[
\frac{1}{\sqrt{f}} = 3.48 - 1.7372 \ln \left( \frac{\varepsilon}{D_h/2} + \frac{9.35}{Re \sqrt{f}} \right) \quad 5 \leq Re \varepsilon \leq 70
\]  

(5.23)

In the transition flow regime, the friction factor rapidly changes with the Reynolds number. However, the above correlations can be used for transitions flows with some uncertainty.

For the wavy channels, Hesselgreaves [26] recommends using the correlation for planar corrugated wall channel proposed by Oyakawa et al. [28] as,

\[
f = 11.0 Re^{-0.53}
\]  

(5.24)

where \( f \) is the fanning friction factor. The characteristic length for the calculation of Reynolds number is the hydraulic diameter. The performance, however, will be strongly dependent on the details of the channel. The wavelength to width ratio of a typical channel is about 7. However, care must be taken in using Equation (5.24) for semi-
circular wavy channel geometry as it is arrived based on the results of planar corrugated wall channel geometry.

5.4 Preliminary Design Calculations

In what follows, some preliminary design calculations based on the current PCHE design of 10 hot and 10 cold plates with 12 channels in each plate are presented. Figure 5.2 shows the variation of Reynolds number with the mass flow rate for the current PCHE configuration. It is assumed that the total mass flow rate is the same on both hot and cold sides and is uniformly distributed among the channels. Helium properties for the hot and cold sides are evaluated for a system pressure of 3 MPa and at the respective hot and cold inlet temperatures of 900 °C and 540 °C. It can be inferred that for the mass flow rate range of 10 to 80 kg/h, the PCHE can be operated in the laminar and laminar-to-turbulent transition regimes.

![Inlet Reynolds number on the hot and cold sides of the PCHE](image)

Figure 5.2 Inlet Reynolds number on the hot and cold sides of the PCHE
Figure 5.3 shows the frictional pressure drop on both the hot and cold sides of the PCHE for mass flow rates ranging from 5 to 80 kg/h. Again, as mentioned above, the mass flow rate is same on both hot and cold sides and is assumed to be uniformly distributed among the channels and the helium properties are evaluated on both the hot and cold sides for a system pressure of 3 MPa and at respective hot and cold inlet temperatures of 900 °C and 540 °C. For the mass flow rates ranging from 5 to 40 kg/h, the flow is assumed to be laminar and fully developed and Equation (5.12) is used in evaluating the fanning friction factor, whereas for mass flow rates from 45 to 80 kg/h, Equation (5.14) is used in evaluating the fanning friction factor. It is obvious that the maximum pressure drop for the straight channel PCHE is less than 3 % of the total system pressure.

![Figure 5.3 Possible range of pressure drop for the hot and cold side](image)
Figure 5.4 shows the approximate variation of hot and cold outlet temperatures of the PCHE with effectiveness for hot and cold inlet temperatures of 900 °C and 540 °C, respectively. In figure 5.5, the variation of heat load of the heat exchanger with effectiveness for a mass flow rate of 80 kg/h on both sides and hot and cold inlet temperatures of 900 °C and 540 °C, respectively is shown. In both the cases, the heat exchanger is assumed to be a balanced counter flow heat exchanger.

![Figure 5.4](image)

**Figure 5.4** Hot and cold side outlet temperatures variation with effectiveness

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5.5 Conclusions

The preliminary design calculations shown above are intended to indicate the operational flow regimes and the performance bounds of the current PCHE design. Various heat transfer and pressure drop correlations presented in this chapter are applicable for straight channels of semicircular cross section. These correlations will later be used to compare the theoretical results with the experimental and numerical results.
CHAPTER 6

SUMMARY, CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORK

6.1 Summary and Conclusions

The current thesis research work is a part of a larger objective aimed at investigating the design, fabrication, testing, modeling, and optimization of PCHEs for potential applications in VHTRs. As is evident from the preceding chapters, this thesis is devoted only to the design and fabrication aspects of high-temperature printed circuit heat exchangers and the helium test facility. In the current work, two PCHE modules were successfully fabricated from Alloy 617 plates. These will be subsequently tested in the helium test facility for their thermal-hydraulic performance. Some salient conclusions that can be drawn from this research work are:

a) The design of a high-temperature helium test facility is complete and it is currently being built at The Ohio State University. It was designed for non-nuclear applications using ASME B31.3 Process Piping Code and is intended for operation at temperatures and pressures up to 900 °C and 3 MPa, respectively. It will enable testing of PCHEs at temperatures typical of VHTRs, albeit, at pressures lower than that proposed to be realized in VHTRs.

b) Investigation of the high-temperature materials showed that Alloys 617 and 230 are the leading candidate materials for the NGNP project. However, this
investigation was carried out mainly for identifying the materials for the high-
temperature helium test facility and the PCHEs. Dictated by material availability
and economics, Alloy 800HT was used for the test facility piping and PCHE
headers while Alloy 617 was used for the heat exchanger plates.

c) Two PCHE modules were successfully fabricated from Alloy 617 plates. The U.S.
available fabrication capabilities for the PCHEs were fully explored. The
processes related to the design and fabrication of PCHEs, such as the
photochemical etching and diffusion bonding techniques were successfully
confirmed for Alloy 617 plates.

6.2 Challenges Associated With the High-Temperature Helium Test Facility

Designing and constructing a test facility to facilitate operation at high temperatures
and pressures typical of VHTR operating conditions is a challenging task. In what
follows, some of the major challenges involved in the design of the high-temperature
helium test facility that is being constructed at The Ohio State University are presented.
This test facility is intended for non-nuclear applications, i.e., Section III Subsection NH
has not been invoked in its design.

a) Selection of appropriate material selection for the test facility and the heat
exchangers is a major challenge. Various aspects ranging from the high
temperature mechanical properties to the corrosion resistance ability of the
material in the presence of helium impurities have to be considered in the
selection.

b) For the purposes of our test facility, welding qualification procedures are
currently being carried out for welding between similar and dissimilar
materials. These include developing qualification procedures for welding Alloy
617 to Alloys 617 and 800HT and welding Alloy 800HT to Alloys 800HT and SS316.

c) Measurement of various parameters such as pressure, temperature and flow is a challenging task for high-temperature applications as the temperatures are well beyond the operating range of the sensors. For temperature measurement, an Alloy 800HT sheathed thermocouple is employed. For pressure measurement, a combination of high-temperature material fittings and a long length of tubing are being employed to isolate the transducer from the pressure source.

6.3 Recommendations for Future Work

Significant amount of work still needs to be done to establish and confirm the applicability of PCHEs in VHTRs. The tasks that need to be accomplished extend from investigating material suitability to testing thermal-hydraulic characteristics of these heat exchangers under reactor conditions. However, in the following recommendations for future work, emphasis has been more on the thermal-hydraulics aspects of the research. Some of the future issues that will be addressed as a part of this continuing work are:

a) To investigate the heat transfer and pressure drop characteristics of the PCHEs and build an extensive database to develop/improve empirical correlations/models for the PCHE heat transfer and pressure drop as functions of Reynolds number, Prandtl number, other appropriate non-dimensional quantities, PCHE material and geometry.

b) Develop a computational model of the PCHE and benchmark it with the database generated.
c) Developing a wavy channel design to realize improved performance of the PCHE. The performance improvement could be different depending on the flow condition, i.e., laminar or turbulent flow. A design that improves the heat transfer performance without introducing a significant pressure drop is being worked out.

d) To carry out experiments with different working fluids with liquid metal/molten salt/helium-nitrogen mixture on one side and gas on the other side and extend the applicability of PCHEs for other Generation IV designs.

e) To carry out the tensile tests at room temperature and high temperature, fatigue failure tests, and creep tests on sample diffusion bonded specimens.

f) Testing the effect of radiation environment on the mechanical properties of the diffusion bonded heat exchanger.

g) Performing a detailed Finite Element Method stress analysis of the PCHE for severe temperature transients.
BIBLIOGRAPHY


APPENDIX A

THERMODYNAMIC AND TRANSPORT PROPERTIES OF HELIUM
Table A.1  Thermodynamic and Transport Properties of Helium [29]

(273 - 1500° K, 0.1 - 10 MPa)

<table>
<thead>
<tr>
<th></th>
<th>Gas Constant: ( R = 2077.22 \ (\text{J/Kg} \ \text{°K}) )</th>
<th>Uncertainty</th>
</tr>
</thead>
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<tr>
<td></td>
<td></td>
<td>&lt; 0.05%</td>
</tr>
<tr>
<td>2.</td>
<td>Equation of State: ( PV = RT + PB(T) ) Where ( B(T) = C_1 + \frac{C_2}{1 - C_3T} + \frac{C_4}{1 + C_5T} )</td>
<td></td>
</tr>
<tr>
<td></td>
<td>( C_1 = 9.489433 \times 10^{-4} , (\text{m}^3/\text{kg}) )</td>
<td></td>
</tr>
<tr>
<td></td>
<td>( C_2 = 9.528079 \times 10^{-4} , (\text{m}^3/\text{kg}) )</td>
<td></td>
</tr>
<tr>
<td></td>
<td>( C_3 = 3.420680 \times 10^{-2} , (\text{K}^{-1}) )</td>
<td></td>
</tr>
<tr>
<td></td>
<td>( C_4 = 2.739470 \times 10^{-3} , (\text{m}^3/\text{kg}) )</td>
<td></td>
</tr>
<tr>
<td></td>
<td>( C_5 = 9.409120 \times 10^{-4} , (\text{K}^{-1}) )</td>
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<td>3.</td>
<td>Compressibility: ( Z = 1 + \frac{P}{RT} B(T) )</td>
<td>&lt; 1.0%</td>
</tr>
</tbody>
</table>
| 4. | Specific Heat:  
|   | \( C_P = 5193.0 \, (\text{J/ Kg K}) \) | < 0.5% |
|   | \( C_V = 3116.0 \, (\text{J/Kg K}) \) | < 0.5% |
| 5. | Enthalpy: \( H - H_0 = C_P T + \left[ B(T) - T \frac{d}{dT} B(T) \right] P \) | < 1.0% |
| 6. | Entropy: \( S - S_0 = C_P \ln \left( \frac{T}{T_0} \right) - R \ln \left( \frac{P}{P_0} \right) - P \frac{d}{dT} B(T) \) | < 1.0% |
| 7. | Viscosity: \( \eta = 3.953 \times 10^{-7} T^{0.667} \left( \frac{N}{m^2} \right) \)  
|   | \( \sigma_n \) = 1.5% |
| 8. | Sonic Velocity: \( c = Z \sqrt{\gamma RT} \) | < 1.0% |
| 9. | Thermal Conductivity: \( k = 2.774 \times 10^{-3} T^{0.701} \, (W/mK) \)  
|   | \( \sigma_k \) = 2.4% |
| 10. | Prandtl Number: \( Pr = \frac{\eta C_P}{k} = 0.740 T^{-0.014} \text{where } T \text{ in } (K) \) | < 3.0% |
APPENDIX B

ASME (SECTION II PART D) ALLOWABLE DESIGN STRESS VALUES
### Table B.1 Maximum allowable stress values (MPa) for seamless pipe and tube [14]

<table>
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<th>Temperature (°C)</th>
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<td>8.27</td>
<td>6.27</td>
<td>5.93</td>
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<td>982.22</td>
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<td>-</td>
<td>3.03</td>
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</tr>
</tbody>
</table>
APPENDIX C

CHROMATOGRAPHIC ANALYSIS OF HELIUM 6.0 RESEARCH GRADE
To:

Attn: 

Praxair Order Number: 6880050-00
Customer Order Number: 
Customer Reference Number: 
Product Lot Number: 848813319
Product Part Number: HE6.6R-T
Test Report Number: 0819587

CERTIFICATE OF ANALYSIS
Helium 6.0 Chromatographic
Purity 99.9999%

<table>
<thead>
<tr>
<th>Cylinder Serial Number</th>
<th>Analytical Results</th>
<th>Analytical Principle/Instrument</th>
<th>Analytical Uncertainty</th>
</tr>
</thead>
<tbody>
<tr>
<td>W-316247</td>
<td>Oxygen &lt;0.1 ppm</td>
<td>P-Delta F'310</td>
<td>± 3%</td>
</tr>
<tr>
<td></td>
<td>Moisture &lt;0.3 ppm</td>
<td>U-Mecoo Aquavolt</td>
<td>± 2%</td>
</tr>
<tr>
<td></td>
<td>Total &lt;0.1 ppm</td>
<td>Q-Teledyne 4020(Flame Ionization Detector)</td>
<td>± 1%</td>
</tr>
<tr>
<td></td>
<td>Hydrocarbons**</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Nitrogen**</td>
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</tr>
<tr>
<td></td>
<td>Carbon Dioxide**</td>
<td>B-Valco 3000</td>
<td>± 0.1 ppm</td>
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<tr>
<td></td>
<td>Carbon</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Monoxide**</td>
<td>B-Valco 3000</td>
<td>± 0.1 ppm</td>
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<tr>
<td></td>
<td>Hydrogen**</td>
<td>B-Valco 3000</td>
<td>± 0.1 ppm</td>
</tr>
<tr>
<td></td>
<td>Argon**</td>
<td>B-Valco 3000</td>
<td>± 0.1 ppm</td>
</tr>
</tbody>
</table>

Cylinder Pressure @50°F (21°C): 2640 psig
Cylinder Volume: 2640 psig

Valve Outlet Connection: 580
Packing Method: Partial Pressure
Date of Fill: May 12, 2008
Expiration Date: December 31, 2011

*Individual analysis
**Batch analysis
Approved Signer: Quality Assurance Reviewer

This analysis of the product described herein was prepared by Praxair Distribution using instruments subject to calibration by certified using Praxair Reference Materials. Praxair Reference Materials are prepared either by weights traceable to the National Institute of Standards and Technology (NIST), Measurement Canada or by using NIST Standard Reference Materials where available.

Note: All expressions for concentration (e.g., % or ppm) are for gas phase, by volume (e.g., ppm) unless otherwise noted.

Way to Analytical Principle:
A. Gas Chromatography with Flame Ionization Detector
B. Gas Chromatography with Electrochemical Detector
C. Gas Chromatography with Flame Photometric Detector
D. Gas Chromatography with Flame Ionization Detector
E. Gas Chromatography with Flame Photometric Detector
F. Gas Chromatography with Ultraviolet Detector
G. Gas Chromatography with Mass Spectrometer
H. Ion Chromatography
I. Ion Chromatography
J. Ion Chromatography
K. Gas Chromatography with Ultraviolet Detector
L. Ion Chromatography
M. Ion Chromatography
N. Mass Spectrometry - ESI or GC/MS
O. Mass Spectrometry
P. Ultraviolet Spectrophotometer
Q. UV Spectrophotometer
R. Spectrophotometer
S. Spectrophotometer
T. Gas Chromatography

IMPORTANT: The information contained herein has been prepared as your request by personnel within Praxair Distribution. While we believe the information is accurate within the limits of the analytical methods employed and is complete to the extent of the specific analyses performed, we make no warranty or representation as to the suitability of the use of the information for any particular purpose. The information is offered with the understanding that any use of the information is at the user's risk. The user is advised to perform his own analysis to insure that the information is adequate for his intended use.

Page 1 of 1 (Lot No. 848813319)