Action of Cryogenic chill on Mechanical properties of Nickel alloy Metal Matrix Composites

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Abstract. In the area of material science engineering, metallurgists may be at the forefront of new technologies, developing metals for new applications, or involved in the traditional manufacture. By doing so it is possible for metallurgist to apply their knowledge of metals to solve complex problems and looking for ways to improve the mechanical properties of the materials. Therefore, an investigation in the present research was made to fabricate and evaluate the microstructure and mechanical properties of composites developed using cryogenically cooled copper chills, consisting of nickel alloy matrix and garnet particles as the reinforcement. The reinforcement being added ranges from 3 to 12 wt. % in steps of 3%. A stir casting process was used to fabricate the nickel base matrix alloy fused with garnet reinforcement particle. The matrix alloy was melted in a casting furnace at around 1350°C, the garnet particulates which was preheated to 600°C, was introduced evenly into the molten metal alloy. An arrangement was made at one end of the mould by placing copper chill blocks of varying thickness brazed with MS hallow block in which liquid nitrogen was circulated for cryogenic effect. After solidification, the composite materials thus synthesized were examined for microstructural and mechanical properties as per ASTM standards.

Keywords: Nickel alloy, Garnet, Cryogenic, Stir casting, Mechanical properties.

1. Introduction
Nickel in elemental form or alloyed with other metals and materials has made significant contributions to our present-day society and promises to continue to supply materials for an even more demanding future. Nickel is a versatile element and will alloy with most metals. Because of their ability to withstand a wide variety of severe operating conditions involving corrosive environments, high temperatures, high stresses, strength, toughness, metallurgical stability, fabricability, weldability, and combinations of these factors nickel and nickel alloys are used for a wide variety of applications [1-5]. The majority of which involve corrosion resistance and/or heat resistance, aircraft gas turbines, steam turbine power plants, and nuclear power systems, chemical and petrochemical industries.

The demand for such functional material to provide high performance has resulted in continuous attempts being made particularly in areas of alloy design and the use of novel processing techniques to develop composites material as serious competitors to the traditional engineering alloys [6-11]. Composite can be said to be a multi-functional system that provides characteristics not obtainable
from any discrete material. Compared with unreinforced metals, MMCs offer designers many benefits, as they are particularly suited for applications requiring higher specific strength and stiffness, higher operating temperature, and greater wear resistance, as well as the opportunity to tailor these properties for a particular application. Metal matrix composites (MMCs) usually consist of a low-density metal, reinforced with particulate or fibers of a ceramic material, such as silicon carbide or graphite. In particular, the particle-reinforced MMCs have been the most popular MMCs over the last two decades in that they exhibit near-isotropic properties by comparison with the continuously reinforced matrices [12]. The combination of properties offered by particle-reinforced nickel metal-matrix composites makes these materials attractive for applications in the marine, aerospace, defence and plumbing industries [13-17].

It is well known that Ni alloys that freeze over a wide range of temperature are difficult to feed during solidification. The dispersed porosity caused by the pasty mode of solidification can be effectively reduced by the use of cryogenic chills. Chills extract heat at a faster rate and promote directional solidification. Therefore, chills are widely used by foundry engineers for the production of sound and quality castings [18-21]. With the increase in the demand for quality composites, it has become essential to produce nickel alloy composites that are free from solidification defects. Nickel alloy castings widely used in automobiles and industries are prone to unsoundness in the form of micro-shrinkage. The primary and most important factor to consider in casting is solidification shrinkage because it contributes significantly to the problems encountered during the feeding of castings. Therefore, cryogenic chill acts as a steep temperature gradient in desired direction and in desired location. As a consequence of using chills, the solidification conditions are altered and so are the casting properties. The ability of the cryogenic chill to extract heat from the molten metal during freezing of the casting is dependent on the size of the chill and thermo-physical properties of the chill material [22-25].

2. Experimental details

2.1. Material Selection

2.1.1. Matrix Material
The ASTM A 494 M grade nickel base alloy was selected has matrix material. The chemical composition, physical and mechanical properties of the selected matrix material are given in the table 1 and 2.

Table 1. Chemical composition of matrix material (ASTM A 494 M grade Ni-alloy) Inconel-625

| Elements                  | % by wt. |
|---------------------------|----------|
| Nickel                    | 58.0 min.|
| Chromium                  | 20.0 - 23.0|
| Iron                      | 5.0      |
| Molybdenum                | 8.0 - 10.0|
| Niobium (plus Tantalum)   | 3.15 - 4.15|
| Carbon                    | 0.10     |
| Manganese                 | 0.50     |
| Silicon                   | 0.50     |
| Phosphorus                | 0.015    |
| Sulfur                    | 0.015    |
| Titanium                  | 0.40     |
| Cobalt                    | 1.0      |
Table 2. Physical and Mechanical properties of base alloy (Matrix material)

| Matrix material | Density (g/cm³) | Melting point in °C | UTS (MPa) | HBW | Yield Strength (MPa) | % elongation |
|-----------------|-----------------|---------------------|-----------|-----|---------------------|-------------|
| ASTM A 494 M    | 8.44            | 1290 - 1350         | 485       | 163 | 275                 | 25          |

2.1.2. Reinforcement
Reinforcement material selected was Garnet, a group of silicate minerals which is one of the hardest naturally available ceramic material [26-27]. The chemical composition, physical and mechanical properties of the selected garnet are given in Table 3 and 4.

Table 3. Chemical composition of Garnet (Almandine - Fe₃Al₂Si₃O₁₂)

| Elements                  | Aluminium Oxide | Iron Oxide | Calcium Oxide | Magnesium Oxide | Titanium Oxide | Silica |
|---------------------------|-----------------|------------|---------------|-----------------|----------------|--------|
| % by wt                   | 19.0            | 34.10      | 3.58          | 4.51            | 2.80           | 35.90  |

Table 4. Physical and Mechanical properties of reinforcement particle

| Reinforcement particle    | Specific gravity | Melting point in °C | Mohr’s Hardness | Refractory Index |
|---------------------------|------------------|---------------------|-----------------|-----------------|
| Almandine garnet          | 4.3              | 1140 - 1280         | 7 - 7.5         | 1.83            |

2.2. Metal matrix Composite preparation
A stir casting process was used to fabricate the nickel base matrix alloy fused with Garnet has reinforcement particle varying from 3 wt.% to 12 wt.% in steps of 3 wt.% for the preparation of metal matrix composites. The matrix alloy was melted in a casting furnace at around 1350°C shown in Figure1 (a). At the same time, the garnet particulate preheated in another furnace set at 600°C for approximately 2 hour to remove surface impurities and assist in the adsorption of gases. Then the preheated 3 Wt. % of garnet particulates, was introduced evenly into the molten metal alloy. This process was repeated for 6, 9 and 12 Wt. % reinforcement. Simultaneously, the molten metal was well agitated by means of a manual mixing using Graphite stirrer, which was carried out for about 5 min. Moulds were prepared using silica sand with 5% bentonite as binder and 5% moisture according to American Foundry men Society (AFS) standards, and were dried in an air furnace. The moulds prepared were rectangular bar shaped ingots of dimensions 150 x 40 x 25 mm as per ASTM standards. A chill blocks were placed adjacent to one end of the mould, the arrangement of sand moulds and chill blocks is shown in figure 1 (b). In addition, the arrangement was made in chill blocks to circulate the liquid nitrogen in and out for cryogenic effect. The chill blocks were made up of copper material of thickness 10mm, brazed with hallow MS blocks of size 150x25x35mm shown in the Figure 1. (c). The molten composite material was next poured into a foundry sand mold prepared, before, after pouring and while pouring of the molten mixture into the mould, liquid nitrogen is poured into the passage provided in hallow steel block. After solidification, the specimens are removed out of the mold. The samples are prepared for testing as per ASTM standards from the specimen taking from the chill end side. The above same procedure was repeated for copper end chill thickness of 20 & 25 mm. The same type of mould was also used to sand-cast a specimen in which case no chill was used.
3. Testing of Composites

3.1. Tensile Strength test
To study the tensile behaviour and to determine the ultimate tensile strength of the matrix composites, specimens were prepared and tested as per ASTM E8M standard as shown in Figure 2 (a). The specimens were machined and prepared using wire cutting as shown in Figure 2 (b). Tensile test was performed using Universal Testing Machine model: TUE-C–400 of 40 T. The machine is a Computer control operating system, which enhances the performance of the machine, uniform and accurate control of the testing, on line display of load vs deformation or stress vs strain, customized test reports, storage and retrieval of test data, rotary encoder detects yield point based on 0.2% proof stress This machine is comply with Grade A of BS 1610 – 1964 and Grade 1 of IS 1828-1991 with an accuracy of +/- 1% of capacity of the machine.

3.2. Hardness test
The hardness testing was carried out for all polished composite specimens prepared from the developed metal matrix composite – Chill end side. The hardness of the specimen determined by Brinell hardness testing machine with 250 kg load and 5 mm diameter steel ball indenter. The indetention time for the hardness measurement was one minute. The tests were carried out at three different locations taken from chill end side of the composite specimen. Each hardness result was obtained from an average of at least three repetitions on the same sample. Meta test model MRB 250 is used for Brinell hardness testing. This is suitable for hardness testing metals and alloys of all kinds, hard, soft, flat round etc. It conforms to IS 2281 – 1983, BS 240 and ASTM E 10 standard.
3.3. Micro Structural Studies
Micro Structural Studies were carried out using metallurgical microscope, NIKON – Japan, Model ECLIPSE LV 150 with the magnification range between 50X – 1000X with computer interfaces which in turn connected to a high resolution Clemax CCD Camera for capturing the micro photographs for micro study & image analysis with the high end CLEM complete software. The specimens for the microstructure analysis were selected from the desired location of the chill end, polished and etched as per ASTM E3-11 standards.

4. Results and Discussion

4.1. Micro Structure result

![Image](a)

![Image](b)

![Image](c)

![Image](d)

![Image](e)
4.1.1. Discussion

It is observed from the above set of microstructure studies shown in figure 3. With reference to the base alloy microstructure, the matrix alloys are uniformly distributed in reinforced particles. This is due to the stirring action and density difference between matrix material and the reinforcement. And the cryogenic effect of passing liquid nitrogen through chills during solidification caused stronger bonding between the matrix material and the reinforcement with very limited clusters, matrix material interfacial integrity, improved grain refinement with minimum porosity. In addition, microstructure reveals that the wettability was good between the particles because of cryogenic cooling of chill. The cryogenic effect has led to improved mechanical properties of the chilled MMC’s as compared with the unreinforced matrix alloy, which is discussed in the later part of the paper. Finally, microstructure reveals fine grain structure because of cryo-chilling effect. It is also observed from the microstructure study that without cryo- chill which gave rise to coarse structure with reduced hardness which is discussed in the later part of the paper.
4.2. Tensile Strength result

Table 5. UTS in N/mm² of cryo-chilled reinforced metal matrix cast using copper chills of varying thickness.

| Chill thickness in mm | Nickel alloy | 3 wt.% of Garnet | 6 wt.% of Garnet | 9 wt.% of Garnet | 12 wt.% of Garnet |
|-----------------------|-------------|------------------|------------------|------------------|------------------|
| 10                    |             | 493              | 514              | 555              | 542              |
| 20                    |             | 498              | 518              | 575              | 560              |
| 25                    |             | 520              | 553              | 635              | 598              |
| No chill              |             | 490              | 499              | 542              | 530              |

Figure 4. Tensile strength of Nickel based composite with varying chill thickness and % Garnet reinforcement

4.2.1. Discussion

The tensile testing of the developed composite results in the table 5 shows the UTS measured by considering the specimen from the chill end for Nickel matrix/reinforced composites cast using cryogenic chills of different thickness. As the chill thickness increases, UTS also increases confirming that the volumetric heat capacity (VHC) of the chill along with liquid nitrogen significantly enhances the UTS. As the reinforcement content is increased; the tensile strength is also increases. The increase in strength is due to the presence and uniform distribution of garnet reinforcements, which is having inferred high strength. Also because of the grain structure obtained from the cryogenic chilling.

In the graph shown in Figure 4, ultimate tensile strength of different cast composites with varying chill thickness and without chill material. UTS of no chill cast composite are lower than that of the remaining dispersoid cast composite with varying chill thickness. Result shows that tensile strength is highest for the 9% dispersoid cast with 25mm chill thickness, followed by those cast with a chill of 10mm and 15mm thickness. From the graph, it is understood that the UTS of the composite increases as dispersoid content is increased up to 9% beyond which in 12% dispersoid the trend reverses.
4.3. **Hardness test result**

**Table 6.** HBW [Brinell hardness] of Cryo-chilled reinforced metal matrix cast using copper chills of varying thickness.

| Chill thickness in mm | Nickel alloy 3 wt.% of Garnet | 6 wt.% of Garnet | 9 wt.% of Garnet | 12 wt.% of Garnet |
|-----------------------|-------------------------------|------------------|------------------|-------------------|
| 10                    | 210                           | 213              | 218              | 214               |
| 20                    | 212                           | 216              | 220              | 218               |
| 25                    | 216                           | 219              | 233              | 225               |
| No chill              | 205                           | 208              | 215              | 210               |

**Figure 5.** Brinell hardness number of Nickel based composite with varying chill thickness and % Garnet reinforcement

4.3.1. **Discussion**

The result in the table 6 shows the Brinell hardness (HBW) value obtained for varying chill thickness and dispersoid content. Like UTS, HBW also increases as the chill thickness increases. This once again confirms that the VHC of the chill enhances not only the UTS but also the BHN. It is shown in Figure 5, hardness of composite with various wt% of dispersoid content and chill thickness. The percentage of reinforcement content is increased along with varying chill thickness, the hardness value of composite are also increased. But compared with no chill cast composite the hardness value is higher than the cryogenic chill cast composite specimens. This significant increase in the hardness can be attributed primarily to presence of harder garnet ceramic particulates in the matrix compared to the hardness of the matrix alloy 163 HBW shown in table 2. A higher constraint to the localized deformation is achieved during indentation due to the presence of reinforcement added and the reduced grain size due to cryogenic chilling.
5. Conclusions
1. ASTM A 494 M grade Nickel matrix alloy and garnet reinforced composites were successfully cast by stir casting route using varying thickness of cryogenically cooled copper chill material. From the analysis of the cast specimens, the following conclusions can be revealed.
2. Microstructure analysis showed the grain refinement, uniform distribution of the reinforcement with minimum porosities.
3. Fine grain structure, uniform distribution of dispersoid and good bonding between the matrix and the dispersoid are obtained with the use of cryogenic chill.
4. Mechanical property characterization of composite cast using 10mm, 15mm and 25mm thick copper chill block containing 3 to 12 Wt.% reinforcement revealed that the presence of garnet particulates in nickel matrix has significantly improved hardness by 14% and strength by 13% (in case of 25 mm copper end chill thickness).
5. Compared to no chill cast composite there is significant increase in mechanical property of the cryo chill cast composite specimen.
6. It was found that mechanical properties improve with increase in dispersoid content up to 9 wt%. beyond which in 12% dispersoid the trend reverses.
7. Volumetric heat capacity (VHC) of the chill and the cryogenic effect are found to increase the amount of heat absorbed.
8. Finally test result showed that these MMCs were greatly influenced by the dispersoid and cryogenic effect in chill blocks. Hardness & UTS of the composite are found to depend on the wt. % of the dispersoid and thickness of chilling. Effect of heat capacity of cryogenic chill is highly dependent on the chilling rate and chill thickness.

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