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Effect of Cryogenic Chill on Mechanical Properties of ASTM A 494 M Grade Nickel Based Alloy Metal Matrix Composites

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An investigation in the present research was made to fabricate and evaluate the microstructure and mechanical properties of metal matrix composites developed using cryogenically cooled copper chills, consisting of ASTM A 494 M grade nickel alloy matrix and garnet particles as the reinforcement. The particle's amount added ranges from 3 wt. % to 12 wt. % in steps of 3%. A stir casting process was used to fabricate the composite. The matrix alloy was melted in a casting furnace at around 1350˚C, the garnet particulates preheated to 600˚C, were introduced into the molten metal alloy. When pouring melt into mould, 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 simultaneously for cryogenic effect. After solidification produced composite materials thus synthesized were examined for microstructural and mechanical properties as per ASTM standards.

Keywords: *Nickel alloy, Chill casting, Garnet, Cryogenic, 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 can be alloyed with most metals. Nickel and nickel alloys are used for a wide variety of applications because of their ability to withstand a wide variety of severe operating conditions involving corrosive environments, high temperatures, high stresses, strength, metallurgical stability, fabricability and weldability[1-2]. The majority of applications require high corrosion resistance and/or heat resistance for aircraft gas turbines, automotive exhaust valves, 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 the areas of alloy design and the use of novel processing techniques to develop composite materials as serious competitors to the traditional engineering alloys [3-10]. Composite is a multi-functional system that provides characteristics not obtainable from any discrete material. Compared with unreinforced metals, metal matrix composites (MMCs) offer designers many benefits as they are particularly suited for applications requiring higher specific strength, stiffness and higher operating temperature.

In particular, the particle-reinforced MMCs have been the most popular among other composite materials over the last two decades, because they exhibit near-isotropic properties in comparison with the continuously-reinforced matrices[11]. The combination of properties offered by particle-reinforced nickel metal-matrix composites makes these materials attractive for applications in the marine, aerospace, and automotive industries.

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[12-17]. 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 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 specific location. As a consequence of using chills, the solidification conditions are altered, influencing 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[18,19].

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2. Experimental details

2.1. Material Selection

2.1.1. Matrix Material

The chemical composition of the selected "ASTM A 494 M grade nickel base alloy" matrix material is given in the Table 1.

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

Elements	% by wt.
Nickel	Balance
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
Aluminum	0.40
Titanium	0.40
Cobalt	1.0

2.1.2. Reinforcement

Reinforcement material selected was Garnet, a group of silicate minerals, which is one of the hardest naturally available ceramic material [20-21].The chemical composition of the selected garnet is given in the Table 2.

Table 2: Chemical composition of Almandine Garnet ($Fe_3Al_2Si_3O_{12}$)

Elements	% by wt.
Aluminium Oxide	19.0
Iron Oxide	34.10
Calcium Oxide	3.0
Magnesium Oxide	4.51
Titanium Oxide	2.80
Manganese Oxide	0.58
Silica	35.90

2.2. Metal Matrix Composite Preparation

A stir casting process was used to fabricate the nickel base matrix alloy fused with Garnet, having reinforcement particles varying from 3wt. % to 12wt. % in steps of 3wt% for the preparation of metal matrix composites. The matrix alloy was melted in a casting furnace at around 1350˚C shown in Figure 1. At the same time the garnet particulate was 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, were 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.

Figure 1: Casting furnace.

The moulds were prepared using silica sand with 5% bentonite as binder and 5% moisture according to American Foundry Society (AFS) standards, and were dried in an air furnace. The moulds prepared were rectangular bar shaped ingots of dimensions 150 × 40 × 25 mm as per ASTM standards. A chill block was placed adjacent to one end of the mould. The arrangement of sand moulds and chill blocks is shown in Figure 2. Also the arrangements were made in chill blocks to circulate the liquid nitrogen in and out for cryogenic effect. The chill blocks placed were made up of copper of thickness 10mm, brazed with hallow MS blocks of size 150x35x40mm are shown in the Figure 3. The molten material at 1350 ˚C was next poured into the sand mold. Liquid nitrogen was introduced into hallow steel block before and after pouring of the molten mixture for cryogenic effect. The above same procedure was repeated for chill thickness of 20 and 25 mm. The same type of sand mould was also used to cast a specimen without chilling effect.

2.3. Characterization and Tests

Microstructural studies were carried out, using optical microscope, Nikon model Eclipse LV 150. The specimens, prepared from the produced composite for the microstructural analysis were selected from the desired location of the chill end, which are polished and etched as per ASTM E3-11 standards[22].

The microstructural and chemical compositions of the samples were also analysed by Zeiss Scanning Electron Microscope (SEM) and its Energy Dispersive Spectrometer (EDS).

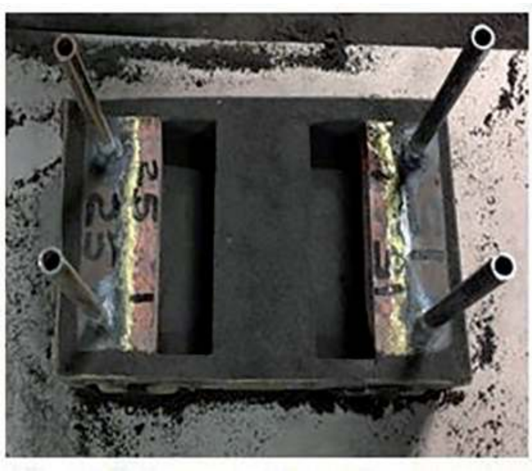

Figure 2: Sand mould with Copper end chill and arrangements for passing liquid nitrogen.

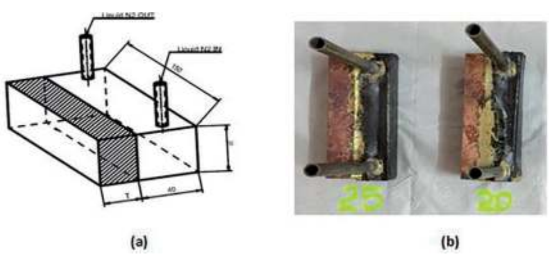

Figure 3: (a), (b) - Brazed copper chill with steel hollow block for passing liquid nitrogen.

To study the tensile behaviour and to determine the ultimate tensile strength of the matrix composites, specimens were prepared and tested as per ASTM E8 / E8M-15a [23] standard as shown in Figure 4. The specimens were machined using wire cutting. Tensile test were performed using Universal Testing Machine model: TUE-C –400.

Figure 4: Tensile test specimens

The Brinell hardness testing was carried out for all polished composite specimens prepared from the developed metal matrix composite as per ASTM E10-15a [24] standard. The hardness of the specimen determined by Brinell hardness testing machine with 250 kg load and 5 mm diameter steel ball indenter. The detention time for the hardness measurement was 1 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.

3. Results and Discussions

3.1. Microstructural Studies

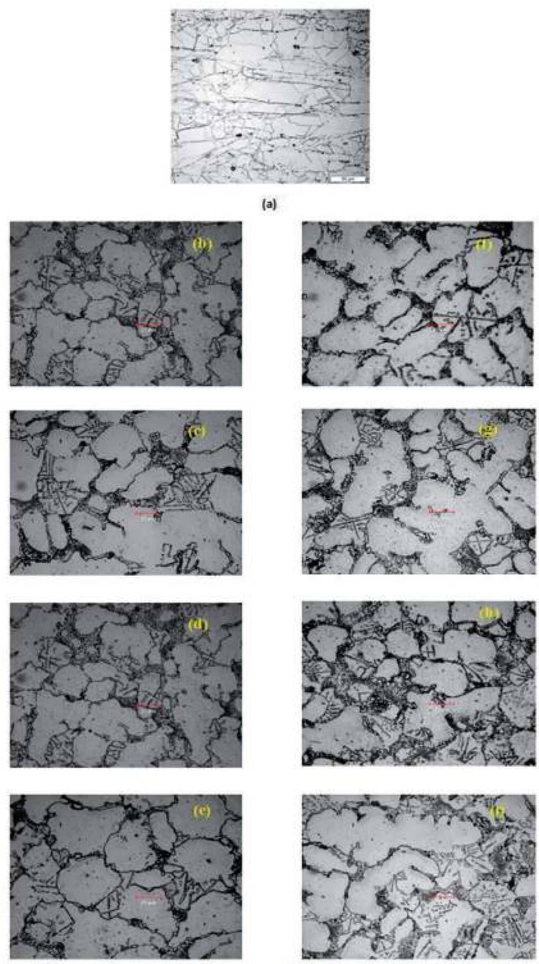

Figure 5. Microstructure images of (a) Base matrix alloy, (b) 3% Garnet with chill, (c) 3% Garnet with No chill, (d) 6% Garnet with chill, (e) 6% Garnet with No chill, (f) 9% Garnet with chill, (g) 9% Garnet with No chill, (h) 12% Garnet with chill, (i) 12% Garnet with No chill.

3.2. SEM Analysis

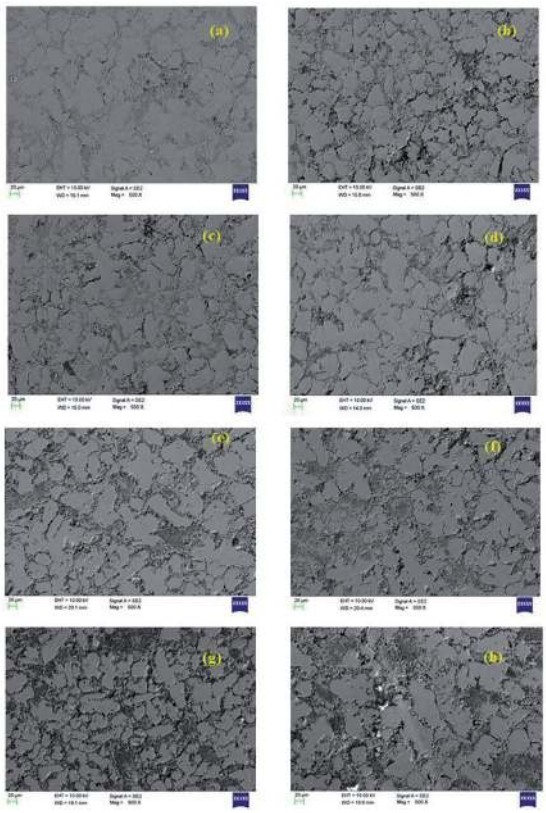

Figure 6. SEM images of (a) 3% Garnet with chill, (b) 3% Garnet with No chill, (c) 6% Garnet with chill, (d) 6% Garnet with No chill, (e) 9% Garnet with chill, (f) 9% Garnet with No chill, (g) 12% Garnet with chill, (h) 12% Garnet with No chill.

3.3. Chemical Composition by EDS Test

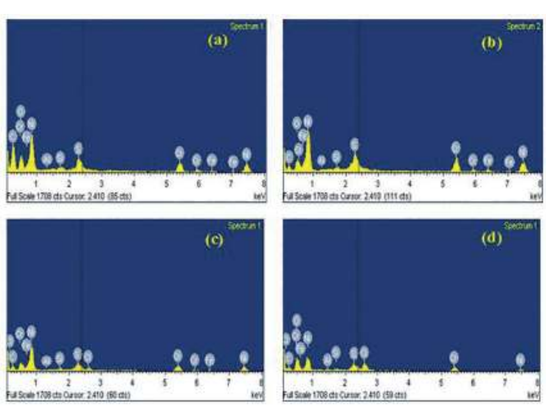

Figure 7. Chemical composition by EDS of (a) 3% reinforcement, (b) 6% reinforcement, (c) 9% reinforcement, (d) 12% reinforcement.

Table 3: Chemical composition by EDS of (a) 3% reinforcement, (b) 6% reinforcement, (c) 9% reinforcement, (d) 12% reinforcement.

Element	Weight 3%	Weight 6%	Weight 9%	Weight 12 %
C K	2.9	1.27	2.17	2.5
O K	5.66	5.95	6.47	2.72
Al K	0.57	1.19	1.71	2.28
Si K	1.28	1.85	2.75	3.66
S K	1.91	2.41	1.53	1.2
Cr K	20.33	19.68	19.08	20.02
Fe K	5.53	6.63	7.2	7.9
Ni K	61.82	61.02	59.09	59.72
Totals	100.00	100.00	100.00	100.00

3.4. Tensile Strength

Table 4. Mechanical properties of matrix material

Matrix material	UTS (MPa)	BHN	Yield Strength	% elongation
ASTM A 494 M	485	163	275	25

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

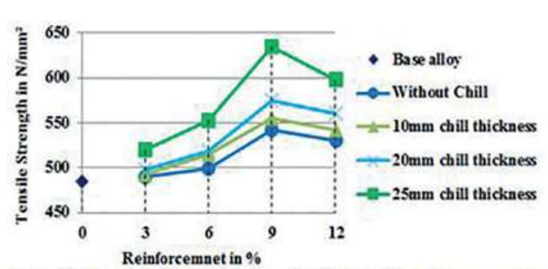

Figure 8: Tensile strength of Nickel based composite with varying chill thickness and % Garnet reinforcement.

3.5. Hardness Test

Table 6: BHN [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

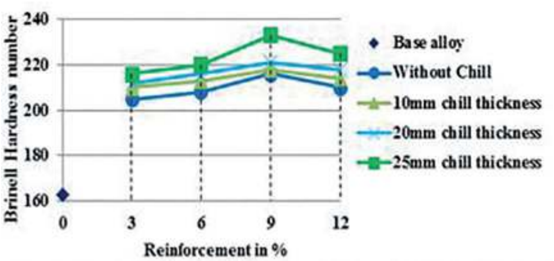

Figure 9: Brinell hardness number of Nickel based composite with varying chill thickness and % Garnet reinforcement.

3.6. Discussions

It is observed from the optical micrographs shown in Figure 5. 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 made the particles to segregate uniformly without allowing time to settle down. 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, and improved grain refinement with minimum porosity. Bonding is perfect between matrix and reinforcement due to preheating of the garnet particle. Micro porosity is also not observed in the microstructure due to cryogenic effect. Finally, microstructure reveals fine grain structure which is observed through scanning electron microscopy shown in Figure 6. And also Figure 7 shows the chemical compositions of developed nickel alloy and garnet reinforced particles analysed through EDS. Table 3 illustrate the EDS analysis data which strongly suggest that the compositions of ASTM A 494 M grade nickel alloy and varying 3 -12 wt. % garnet particles.

The tensile testing of the developed composite result in the Table 5 shows the ultimate tensile strength (UTS) measured by considering the specimen from the chill end for Nickel matrix/reinforced composites cast of different thickness. It is observed that the tensile strength of no chill cast composite are lower than that of the remaining chill cast composite with varying chill thickness. As the chill thickness increases, UTS also increases confirming that the volumetric heat capacity (VHC) of the copper chill along with liquid nitrogen significantly enhances the grain structure. As the reinforcement content is increased; the tensile strength is also increases. The increase in tensile strength is due to the presence and uniform distribution of garnet reinforcements which is having inferred high strength, and grain structure obtained from the cryogenic chilling. UTS of different cast composites with varying reinforcement weight percentage and chill thickness is shown in Figure 8. The result shows that tensile strength is increasing up to 9 wt. % Garnet content and then start gradually decreasing for 12 wt. %, where the trend is reverse.

The result in the Table 6 shows the Brinell hardness (BHN) value obtained for varying chill thickness and Garnet content. Like UTS, BHN 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. Compared with no chill cast composites the hardness value is higher in the cryogenic chill cast composite specimens. This significant increase in the hardness can be attributed primarily due to presence of harder garnet particulates in the matrix alloy with reference to the hardness of the matrix alloy 163 BHN shown in Table 4. From the graph shown in Figure 9 the hardness values demonstrate the pattern that as the percentage of Garnet is increased from 3 wt. % to 9 wt. % there is an increase in the composite's hardness. Later there is a decrease in the hardness when garnet substance is extended to 12 wt. %.

4. Conclusion

(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) Microstructural analysis using optical micrograph and SEM showed the grain refinement, fairly uniform distribution of the added Garnet particulates with minimum porosities.

(3)Uniform distribution of added Garnet particles and good bonding with the matrix alloy is obtained because of the stirring action.

(4)Fine grain structure and soundness of the composite is dependent on cryo chilling effect.

(5) 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 tensile strength by 13% (in case of 25 mm copper end chill thickness).

(6) Compared to no chill cast composite there is significant increase in mechanical property of the cryo chilling cast composite specimen.

(7) It was found that mechanical properties can be improved with increase in Garnet particles content up to 9 wt. %, beyond which at 12 wt. % the trend reverses.

(8) It is clearly indicated by EDS test that the Garnet content is distributed in right proportion in the matrix alloy.

(9) Finally, the test result showed that these MMCs were greatly influenced by the Garnet particles addition and cryogenic effect. Hardness and 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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