Influence of fused Silica and chills incorporation on Corrosion, Thermal and Chemical composition of ASTM A 494 M Grade Nickel alloy

G Purushotham¹ and Joel Hemanth²

¹Department of Aeronautical Engineering, Mangalore Institute of Technology & Engineering, Moodbidri, Karnataka, INDIA 574 225.
E-mail: purushotham68@gmail.com

²Department of Mechanical Engineering, HMS Institute of Technology, Tumkur Karnataka, INDIA 572104.

ABSTRACT
A review of a host of relevant literature on the composites leads to some important observations on the gap that prevails for developing the composite with increased strength to weight ratio, improved thermal properties and reduced corrosion rate with the addition of fused SiO₂ dispersoid for the nickel based alloy. In the arena of engineering, metallurgists look for techniques to improve the thermal, corrosion and chemical properties of the materials. In this connection an investigation has been carried out to fabricate and evaluate the corrosion, chemical and thermal properties of chilled composites consisting of nickel matrix with fused silica particles (size 40–150 μm) in the matrix. The main objective of the present research is to obtain fine grain Ni/SiO₂ chilled sound composite having very good properties. The dispersoid added ranged from 3 to 12 wt. % in steps of 3%. The subsequent composites cast in molds containing metallic and non-metallic chill blocks (MS, SiC & Cu) were tested for their microstructure, chemical, thermal properties and corrosion behavior.

Keywords: Chills, Corrosion, Fused silica, Metal matrix composite, Nickel alloy, SEM & EDX.

1. INTRODUCTION

Many researchers reported the advantages of nickel alloy compared to other materials including the potential for high hardness, good abrasion resistance, improved corrosion resistance and micro creep performance. Furthermore, fabrication of the discontinuously-reinforced nickel composite can be achieved by standard metallurgical methods [1-3]. Nickel alloy based metal matrix composites are the class of advanced materials that are well suited for pumps, valves and automotive industries because of their strength, corrosion resistance and for electric and electronic industry because of their high thermal and electrical conductivities [4-6]. In particular, the particle-reinforced Metal Matrix Composites (MMCs) are attractive since they exhibit near-isotropic properties in comparison with the continuously-reinforced matrices.

With the increment in the interest for quality composites, it has become crucial to create nickel combination composites that are free from hardening imperfections. It is understood that Ni alloys solidify over an extensive variety of temperature and are hard to nourish amid cementing. Nickel composite is inclined to surrender as a small scale shrinkage. The scattered porosity brought about by the pale method of hardening can be successfully diminished through the utilization of chills. Chills concentrate heat up at a speedier level and advance the directional solidification. In this manner chills remain broadly utilized by foundry engineers for the generation of comprehensive and excellence
castings. Smaller scale contraction or scattered penetrability in the composite can be reduced by the reasonable area of chills [7-9]. 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. Micro-shrinkage or dispersed porosity in the composite can be minimized by the judicious location of chills. Chills helps to achieve a steep temperature gradient in the desired direction and at the desired location. As a consequence of using chills, the solidification conditions are altered and so are the casting properties [10-13]. The ability of the 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. In other words, the capacity of the chill to absorb heat from the casting is taken as a measure of its efficiency. The Volumetric Heat Capacity (VHC), which takes into account the volume, the specific heat and the density of the chill material has been identified as an important parameter in evaluating the efficiency of the chill and the corrosion resistance of the materials [14-16]. It has been found by certain researchers that the chilling process adopted in casting of composites helps in improving the quality of the castings.

2. EXPERIMENTAL DETAILS

2.1. Material Selection

The metal matrix material was selected available pure nickel base alloy (Monel metal). The chemical composition of the matrix is given in the table 1. The reinforcement was pure SiO$_2$ of particles size 40µm to 150µm. Induction method was used for preparation of the composite.

Table 1. Composition of Nickel alloy (M-35-1)

| Element | Ni | Cu | Fe | C | Mn | Si | P |
|---------|----|----|----|---|----|----|---|
| Composition in wt.% | Bal | 26.6 | 0.513 | 0.196 | 3.02 | 0.8 | 0.0232 |

| Element | S | Cr | Co | Al | Ti | Sn | Pb |
|---------|---|----|----|----|----|----|----|
| Composition in wt.% | 0.0027 | 0.0252 | 0.0826 | 0.112 | 0.121 | 0.0237 | 0.0753 |

Table 2. Properties of matrix alloy and the reinforcement (M35-1 Ni alloy)

| Property | Nickel alloy | Silica |
|----------|--------------|-------|
| Density  | 8.908 g/cc, | 2.78g/cc |
| Tensile Strength | 450 MPa | 48.3 MPa |
| Hardness | 638 MPa | Rc: 89 |
| Crystal structure | FCC | -------- |
| Melting Point | 1455°C | 1650°C |
| Thermal Conductivity | 1.38W/m.°K | 1.3W/m.°K |
| Coefficient of thermal expansion | ------ | 0.4x10^-6/k |

3. PREPARATION OF THE COMPOSITE

The nickel based alloy/fused SiO$_2$ metal matrix composite was manufactured by utilizing the induction furnace along with stirrer for varying the percentage of reinforcing particles starting from 3 wt. % to 12 wt. % in steps of 3 wt. %. This technique is the most practical to create the composite ingredients. The matrix was initially superheated over its melting temperature (1560°C) and preheated SiO$_2$ (600°C)
particulates included in the liquid metal and the liquid metal was blended appropriately. The melt at 1560°C was filled with sand molds. The molds for the plate sort of moldings of size 225*50*25 mm (American Foundry Men Society standard) were prepared utilizing silica sand with 5% bentonite as the binder and 5% moisture and finally dried. These castings were cooled from one end by distinctive metallic and non-metallic chill blocks set in the mold.

![Induction Furnace](image1)

![Chills arrangement](image2)

1) SiC chill 2) Cu chill 3) MS chill 4) Without chill

![Pure Nickel planks](image3)

![Fused Silica](image4)

![Mechanical stirrer](image5)

![casted specimens](image6)

**Figure 1.** Shows the Photographs of mold, induction furnace, stirrer and casted specimens
4. TESTING OF COMPOSITES

4.1 Scanning Electron Microscope

Figure 2. Scanning Electron Microscope

The Scanning Electron Microscope (SEM) utilizes an engaged electron test to extricate structural and chemical data point-by-point from a chilled locale of enthusiasm for the specimen. The great resolution of an SEM makes it a capable apparatus to portray an extensive variety of examples at the nanometer to micrometer length scales in the amplification of 1,00,000x can be accomplished. EVO18 can be operated in various modes such as secondary electron (SE), back scattered electron (BSD) and variable pressure (VP) modes. The attached EDS detector helps in quantifying the elements present in the sample.

4.2 Corrosion Test

Figure 3. Tafel Polarisation setup and the specimen for corrosion test

Electrochemical measurements were carried out by using an electrochemical work station, (CH600D-series, U.S. Model with CH instrument beta software). The electrochemical cell used was a conventional three-electrode compartment having glass cell with a platinum counter electrode and a saturated calomel electrode (SCE) as reference. The working electrode was made up of monel metal. All the values of potential were with reference to the SCE as shown in figure 3.
Arrangement of Specimen fixing in TMA/SDTA 1 HT/1600 equipment

Figure 4. TMA/SDTA setup and the specimen for thermal conductivity

All the test specimens for thermal conductivity and coefficient of thermal expansion were prepared by means of a suitable ASTM requirement and as per the equipment TMA/SDTA 1 HT/1600 requirement, specimens were prepared by cutting suitable length thoroughly ground and polished the edge surface of the specimens. Figure 4 shows the samplings for thermal conductivity and coefficient of thermal expansion.

5. RESULTS AND DISCUSSIONS

5.1 Microstructural studies

Base Metal

3% SiO₂ plain
Figure 5. Microstructural studies of base metal, different weight % of SiO2 and different chills
**Discussion:** The microstructures of fabricated as deposited samples, were observed to be in agreement with those predicted microstructures. It is witnessed from the microstructural revisions that the structure was observed to be close packed dendritic with only primary dendrite arms visible and with no evidence of precipitation or grain boundary segregation of solute atoms. Moreover, the dispersoid is spread homogeneously and this is predominantly because of stirring and density differences. Closeness is flawless between matrix and dispersoid owing to rewarming of the dispersoid. Micro porosities have also not been detected in the microstructure.

5.2 *Scanning Electron Micrographs of Fractured Surfaces*

![SEM photograph of base metal (500X)](image)

![Shows the SEM photograph of 3 wt. % SiO2](image)

![SEM photograph of 6 wt. % SiO2 (without chill)](image)

![SEM photograph of 9 wt. % SiO2](image)

![SEM photograph of 6 wt. % SiO2- SiC chill](image)

![SEM photograph of 9 wt. % SiO2-Cu chill](image)

**Figure 6.** SEM Photographs of different wt. % of SiO2 with different chills
In order to understand the mode of fracture for the specimens failed under the tensile test, fractographic analysis was carried out on the composite material developed. The fractomicrographs of 25mm thickness chill cast with Ni/SiO$_2$ containing 3 wt. %, 6 wt. %, 9 wt. % & 12 wt. % SiO$_2$ are shown from figures. Figures show respectable closeness sandwiched between the matrix and the reinforcement. It was observed from the micro photographs that the fractured surfaces on the dispersoid content increase the mode of fracture shifts from ductile to brittle. From figures 3 wt. % shows shallow dipped surface indicating ductile fracture. Figures 6 wt. % shows the transforming from ductile to brittle indicating a bi-model type of fracture and figures 9 wt. % shows a cleavage type of fracture.

5.3 Corrosion Test
Corrosion test results

Figure 7. Graph of current v/s potential of corrosion rate for different wt. % SiO$_2$ of nickel alloy.
Figure 8. Plot of corrosion rate v/s wt. % of SiO$_2$ for the composites cast under the influence of different chills.

Diagram (Figure 8) The corrosion rate of the unreinforced matrix alloy (corrosion rate=2.563 mpy) is higher than that of the composites, because in the former there is no reinforcement phase and the matrix alloy does not have much corrosion resistance to the acid media. Silica is a hard ceramic that stays latent and is itself unpretentious by the acidic media during the tests. By and by, the outcomes demonstrate a change in the corrosion resistance as the fused silica substance is increased in the composite, showing that the intertwined silica particulates impact the consumption attributes of the composites but not electrochemically. It is likewise seen from the chart that the corrosion rate is reduced as the dispersoid is built even up to 12 wt. % for composites cast utilizing distinctive chills.

5.4 Coefficient of thermal expansion
5.4.1 Coefficient of thermal expansion for the addition of reinforcement (SiO$_2$) cast using different chills for different temperatures
5.4.2 CTE for the addition of reinforcement (SiO2) for different temperatures cast using different chills

The main findings from the experiment is that the coefficient of thermal expansion for both nickel alloy (base metal) and nickel alloy with fused silica composites decline with the rise in temperature. The rate of coefficient of thermal expansion drops with a rise in the weight percentage of SiO2 for 200°C.

5.5 Thermal Conductivity

Figure 9. CTE for the addition of SiO2 for different temperatures cast using different chills

Figure 10. Thermal conductivity for the addition of reinforcement (SiO2) for different temperatures cast using different chills
Results of Thermal conductivity:
In the present study of the composite material [from figures] the thermal conductivity has been measured for the specimens of nickel alloy with varying percentages of fused silica in the presence of different chills and for different temperatures.

The main outcome of the experiment is that the thermal conductivity of both the nickel alloy (base metal) and that of the nickel alloy with fused silica composite reduces with the rise in temperature of SiO2 up to 6 wt. %. The rate of thermal conductivity shrinks with the escalation in weight percentage of SiO2 due to the rise of temperature up to 200°C and then the thermal conductivity of the composite material gradually starts increasing with the further rise of temperature. Hence this property of thermal conductivity is most suitable for engineering applications.

5.6 Chemical Composition by EDX Test

| Element | Weight% | Atomic% |
|---------|---------|---------|
| Si K    | 0.83    | 9.73    |
| P K     | 0.73    | 1.34    |
| S K     | 0.20    | 0.51    |
| Mn K    | 2.03    | 2.09    |
| Fe K    | 2.04    | 2.06    |
| Ni K    | 62.17   | 57.04   |
| Cu K    | 30.79   | 26.54   |
| Nb L    | 1.13    | 0.69    |
| **Totals** | **100.00** | **100.00** |

**Base Metal**

| Element | Weight% | Atomic% |
|---------|---------|---------|
| Si K    | 5.63    | 7.42    |
| P K     | -0.02   | -0.04   |
| S K     | 0.00    | 0.00    |
| Mn K    | 2.90    | 3.03    |
| Fe K    | 2.14    | 2.21    |
| Ni K    | 62.07   | 62.70   |
| Cu K    | 26.32   | 24.70   |
| Nb L    | -0.04   | -0.02   |
| **Totals** | **100.00** | **100.00** |

**6% SiO2**

| Element | Weight%  | Atomic% |
|---------|----------|---------|
| Si K    | 11.07    | 11.26   |
| P K     | 0.20     | 0.37    |
| S K     | 0.07     | 0.12    |
| Mn K    | 3.40     | 3.51    |
| Fe K    | 2.30     | 2.33    |
| Ni K    | 61.17    | 60.96   |
| Cu K    | 22.41    | 23.48   |
| Nb L    | -0.06    | -0.04   |
| **Totals** | **100.00** | **100.00** |

**9% SiO2**

| Element | Weight%  | Atomic% |
|---------|----------|---------|
| Si K    | 2.39     | 4.03    |
| P K     | 0.20     | 0.38    |
| S K     | 0.18     | 0.33    |
| Mn K    | 2.20     | 2.32    |
| Fe K    | 1.78     | 1.85    |
| Ni K    | 67.60    | 66.78   |
| Cu K    | 25.66    | 23.42   |
| Nb L    | -0.01    | -0.01   |
| **Totals** | **100.00** | **100.00** |

**12% SiO2**

**Figure 11.** EDX test for different wt. % of SiO2
6. CONCLUSIONS

1. Nickel based metal matrix composite can be casted successfully from a conventional electric induction furnace.
2. Different chill material and the dispersoid content however do significantly affect the physical and chemical properties of the composites.
3. Microstructure of the chilled composites is finer than that of un-chilled matrix alloy with uniform distribution of SiO$_2$ particles. Strong interfacial bond was observed with no agglomeration between the matrix and the dispersoid.
4. It was observed from the micro photographs that the fractured surfaces on the dispersoid content increase the mode of fracture shifts from ductile to brittle. From figures 3 wt. % shows shallow dipped surface indicating ductile fracture. Figures 6 wt. % shows the transforming from ductile to brittle indicating a bi-model type of fracture and figures 9 wt. % shows a cleavage type of fracture.
5. It is clearly indicated that the dispersoid is uniformly distributed in the matrix alloy by EDX test.
6. Chill material and the dispersoid content however do significantly affect the corrosion properties of the composites. It was found that the corrosion resistance increase with an increase in the dispersoid content up to 9 wt. %. Hence this property of corrosion resistance is most suitable for marine applications.

7. The main findings from the experiment is that the coefficient of thermal expansion for both nickel alloy (base metal) and nickel alloy with fused silica composites decreases with the increase in temperature. The rate of coefficient of thermal expansion decreases with the increase in weight percentage of SiO$_2$ by raising the temperature up to 200°C.

7. REFERENCES

[1] Joel Hemanth 2009 Cryo effect during solidification on the tribological of Aluminium- alloy/glass (SiO$_2$) MMC. *Journal of composite materials*, vol.43, No.06.
[2] Pradeep K. Rohatgi 19993 Metal matrix composites, *Defense Science Journal*, vol 43, No:4, pp323-349.
[3] Meenu Srivastava, A Srinivasan, VK William Grips 2011 Influence of Zirconica incorporation on the mechanical & chemical properties of Ni-Co alloys, *American Journal of material science*, 113-122.
[4] KR Marikkannu, K.Anutha, G.Paruthimal Kalaighn & T.Vasudevan 2004 Studies on Ni-Al electrocomposite coatings of over mildsteel substrate. *International symposium of research students on material science & engineering*, Dec 20-22, Chennai.
[5] Ayhan EROL, Ahmet YONETKEN, Ismail Yildiz, Muzaffer ERDOGAN 2009 Fabrication of Ni MMC reinforced with SiO2 by microwave sintering, 5th International Advance Technical Symposium, May 13-15.
[6] K.Funatane & K.Kurasania, P.A.Fabiyi & M.F. Puz 1994 Improved engine performance via use of Nickel ceramic composites (NCC coat), *SAE International* Feb 28-Mar 3.
[7] Zhao Lei, Zhang Lixia, Tian Xiaoyu, He Peng, Feng Jicai 2011 “Interfacial micro structure and mechanical properties of joining electroless Nickel plated Quartz fibres reinforced Silica composite to Invar, *Material and Design*, 32, 382-387.
[8] Kh.A.Ragab, R.Abdel- Karim, S.Farag, S.M.El-Raghy, H.A.Ahmed 2010 “Influence of SiC, SiO$_2$ and graphite on corrosive wear of bronze composites subjected to acid rain, *Tribology International*, 43, 594-601.
[9] J.Mendoza-Canales and J.Martin-Cruz 2008 “Corrosion behavior of Titanium and Ni-Based alloys in HCl and HCl+H$_2$S environments”, *International Journal of Electro Chemical Science*, pp 346-355.
[10] El-Sayed M.Sherif, Asiful H. Seikh 2015 “Effect of Exposure Period and Temp. On the Corrosion of Incoloy Alloy 800TM in Hydrochloric acid Pickling Solutions” *Int. J. Electrochem. Sci.*, 10,1843-1854.
[11] Behnam Sabbaghzadeh, Reza Parvizi, Ali Davoodi, 2014 “Corrosion evaluation of multi-pass welded nickel-aluminium bronze alloy in 3.5% sodium chloride solution: A restorative application of gas tungsten arc welding process”, Materials and Design, 58, 346-356.

[12] Yuhang Hou, Yunping, Chen Zhang, Akihiko Chiba, 2014 “Effect of cold working on corrosion resistance of Co-modified Ni-16Cr-15Mo alloy in hydrofluoric acid solution”, Corrosion science 89, 258-267.

[13] Christopher DellaCorte, Nickel-Titanium alloys: 2010 “Corrosion proof alloys for space bearing, components and mechanism applications”, NASA Kennedy Space Center, May 12-14.

[14] Brunoro G, Frignani A, Colledan A, Chiavari C 2003 “Organic films for protection of copper and bronze against acid rain corrosion”, Corrosion Sci; 45:2219–31.

[15] Andrew T Nelson, Joshua T White, David A Anderson, 2014 “Thermal expansion, Heat capacity and Thermal conductivity of Nickel Ferrite”, The American Ceramic Society, pp1-7.

[16] R. Przeliorz, J. Piatkowski, 2015 “Thermo physical properties of nickel-based cast super alloys”, Metallurgy, 54, 543-546.