

Development of Nickel Alloy Reinforced with Fused SiO₂ Chilled Composites and Evaluation of Thermal Properties (Thermal Conductivity & Coefficient of Thermal Expansion) and Temperature Distribution by Finite Element Analysis (FEA)

Joel Hemanth

Department of Mechanical Engineering, H. M. S. Institute of Technology, Tumkur, Karnataka, India

Email: joelhemanth@hotmail.com, joelhemanth@gmail.com

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Abstract

This paper presents the results obtained and the discussions made from a series of thermal experiments involving Nickel alloy (ASTM A 494 M) reinforced with fused SiO₂, and size of the particles dispersed varies from 80 - 120 µm and amount of addition varies from 3 to 12 wt.% in steps of 3 wt.%. The resulting chilled MMCs are solidified under the influence of copper chill of 25 mm thickness to study the effect of chilling on thermal behaviour. Micro-structural studies indicated that the reinforcement distribution is uniform with very good bonding due to chilling effect. Thermal properties were found to decrease significantly with increase in SiO₂ content in chilled MMCs. It is concluded from the research that reinforcement content and the temperature has an effect on coefficient of thermal expansion and thermal conductivity of the chilled composite developed. Finite element analysis of the exhaust valve of the IC indicates that chilled Ni alloy composite developed in the present research can be used as alternate material for the existing valve material (Ni-Cr alloy steel). All the tests conducted in this research are in conformance with ASTM standards.

Keywords

Nickel, Thermal, Chill, Composite and Conductivity

1. Introduction

Nickel alloy-based metal matrix composites are popular materials that are pri-

marily suited for the manufacture of pump bodies, valves and automotive parts because of their high strength, high corrosion resistance having high thermal and electrical conductivities that are best suited for electrical and electronic industries [1]. The need of such a composite material to provide high qualities has resulted in several researches that are being made particularly in areas of alloy design and the use of conventional processing techniques to develop composites [2] [3]. In general, the particle-reinforced metal matrix composites (MMCs) are attractive since they exhibit near-isotropic properties as compared with the continuously-reinforced composite materials. Several researchers have reported the advantages of Ni alloy that it offers over the other material including a potential for high hardness, wear resistance, improved fracture crack propagation and good micro creep performance [4] [5] [6]. Furthermore, fabrication of the discontinuously reinforced nickel composites can be achieved by standard metallurgical processing techniques and these materials can be processed by using conventional facilities. The properties gained by the combination of Ni alloy and the reinforcement made these materials very attractive for applications in marine, aerospace, defense, pump manufacturing and plumbing industries [7] [8] [9] [10]. Previous researches have been conducted to study and analyze the metallurgical and mechanical properties of nickel and its alloys [11] [12].

It is well known that most of the non ferrous metals including Ni alloys freeze over a wide range of temperature are difficult to feed during freezing. The defects especially the micro porosity caused by the pasty mode of solidification can be effectively reduced by the use of chills. Chills are heat sinks to extract heat at a faster rate to make solidification directional. Therefore chills since a long time ago are widely used by foundry engineers for the production of sound and quality castings. With the increase in the demand for quality composites, it has become essential to manufacture nickel alloy composites that are free from solidification defects [13] [14] [15] [16] [17]. Nickel alloy castings that are widely used in automobile, marine and pump industries are prone to defect in the form of micro-shrinkage or the dispersed porosity. Therefore these in the composite can be eliminated by inserting chills judiciously at desired locations. Such a chill inserted sets up 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 chill to extract heat from the freezing molten metal during solidification of the casting is dependent on the size of the chill, material of the chill and its thermo-physical properties. In other words, the ability of the chill to absorb heat from the casting is taken as a measure of its efficiency. The VHC (Volumetric Heat Capacity), which takes into account the volume, specific heat and density of the chill material, has been identified as an important parameter in evaluating the efficiency of the chill [18].

Literature review reveals that so far very less work has been done on Ni-based composites and no work has been done on thermal properties of chilled Ni composite. Hence the present research is undertaken to fill the void.

2. Experimental Procedure

A detail procedure of melting and composite preparation is described elsewhere by number of researchers [15]. In this investigation, nickel alloy matrix composites are fabricated using chill casting technique using copper chill of 25 mm thick. The fabrication of the composite was carried out by adding the fused silica particles in the range of 3 to 12 wt.% insteps of 3 wt.%. The size of the particulate added ranges from 80 to 120 μm . After melting the matrix material in a furnace at 1600°C in an inert atmosphere, fused SiO_2 particles preheated to 500°C were introduced evenly into the molten metal alloy by means of special feeding attachment. Meanwhile, the reinforcement treated molten nickel was well agitated by means of a mechanical impeller rotating at 400 rpm to create a vortex. The reinforcement treated molten Ni alloy was poured in to the mold cavity which was cooled from one end by a copper chill block (25 mm thick) set in the mold. The molds for the plate type of castings of size 225 × 150 × 35 mm (American Foundrymen Society standard) were prepared using silica sand with 5% bentonite as binder and 5% moisture. Finally molds were dried in an air furnace at a temperature of 600°C. After solidification these test blocks will be subjected to heat treatment (aging) and later test specimens were taken from the chill end for various thermal tests including microstructural scanning electron microscopy studies.

Composition and properties of the matrix material: Nickel alloy, ASTM A 494 M grade.

Nickel: 55%; Cr: 15.5%; Mo: 16%; Mn: 1%; Si: 1% and traces of P, S, etc. Tensile Strength: 500 MPa; Yield strength: 275 MPa; Elongation: 4%; Melting temperature: 1575°C.

Properties of reinforcement (Fused SiO_2):

This special type of reinforcement having very good mechanical and thermal properties (listed below) was obtained from Dupre Minerals, Staffordshire, England.

Density: 2.78 gm/cc, Hardness Rc: 89, Melting point: 1660°C, Young's modulus: 87 GPa.

Microstructural examination was conducted on the polished specimens using NEOPHOT-21 optical microscope.

Finite Element Analysis (FEA) was carried out for the IC engine exhaust valve using the chilled composite developed to analyze the temperature distribution using the SOLID WORKS STATION software by Microsoft.

Thermal Conductivity and Coefficient of Thermal Expansion

All the test specimens for thermal conductivity and coefficient of thermal expansion were prepared according to ASTM standard and as per the standards of the testing equipment TMA/SDTA 1 HT/1600 thermal analysis tester as shown in **Figure 1**. The TMA/SDTA 1 HT/1600 thermal analyzer (for both thermal conductivity and coefficient of thermal expansion) integrates the Swiss meticulousness



Figure 1. Photograph of TMA/SDTA 1 HT/1600 thermal analyzer.

mechanics and is accessible in four types with furnace systems optimized for capacities between RT and 1600°C.

3. Results and Discussions

3.1. EDX Test for Chemical Composition of the Base Metal

To know the reinforcement content and the elements present in the matrix alloy, the EDX test was carried out for different test specimens cast under the influence of Cu end chill block. From **Figure 2** it is observed that the reinforcement content is present in the composites where SiO₂ was varied from wt. 3% to 12 wt.%. It is also observed from the figure that the chemical composition of the matrix alloy was also confirmed. Thus from EDX analysis of the composite developed the presence of silica and other elements present in the matrix was ascertained.

3.2. Microstructural Studies

Microstructural studies were carried for the samples taken near the chill end of the castings. This study was carried out to reveal the dispersion of reinforcement and the effect of chilling on the microstructure of composites. It is observed from the microstructural studies that (**Figure 3** and **Figure 4**) 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. Strong interfacial bond was observed with no agglomeration between the matrix and the reinforcement. Moreover the reinforcement was dispersed homogeneously and uniformly and this is predominantly because of the stirring effect. Micro porosities have also not been observed in the microstructural studies

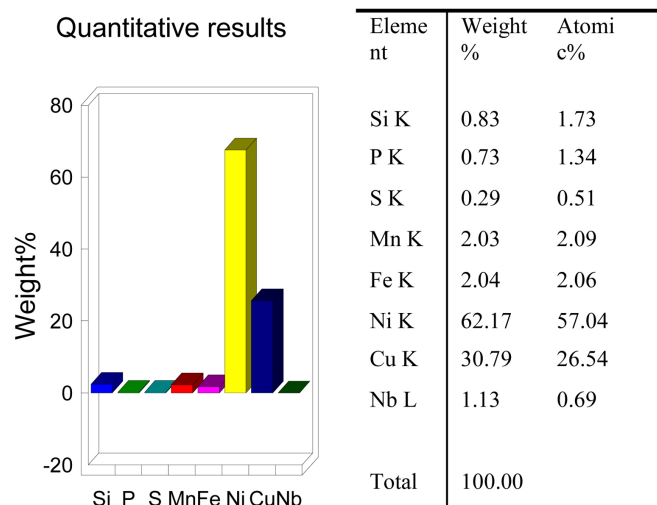


Figure 2. EDAX test results.

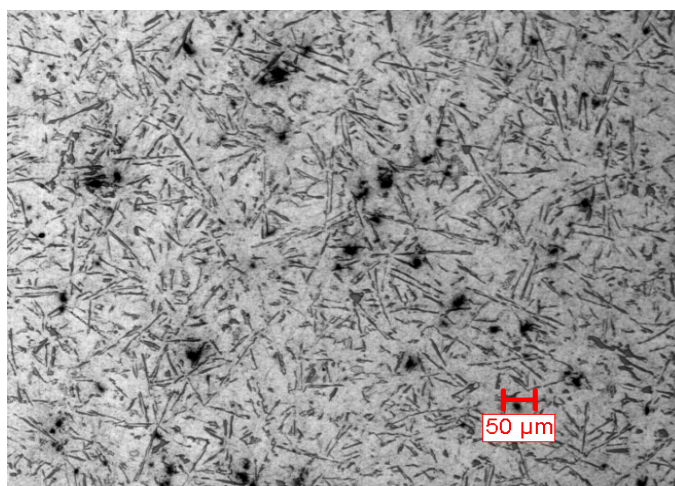


Figure 3. Microstructure of Chilled Ni-MMC containing 9 wt.% reinforcement. (500×, 50 μm magnification).

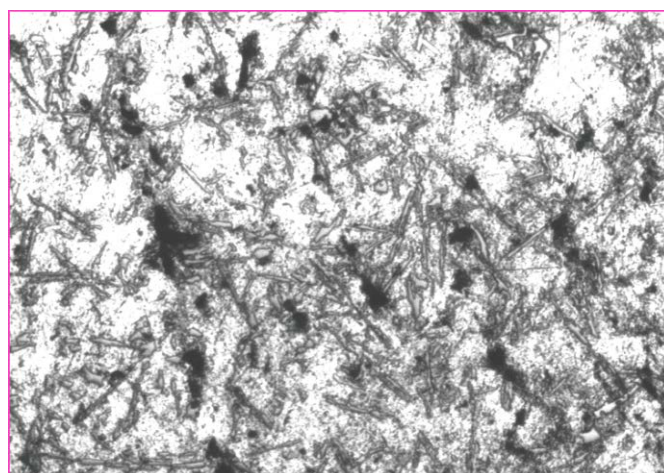


Figure 4. Microstructure of unchilled Ni alloy MMC containing 9 wt.% reinforcement. (500×, 50 μm magnification).

and the microstructure conceals the fine grain structure because of the influence of chilling (**Figure 3**). Whereas the un-chilled Ni composite (**Figure 4**) shows the coarse structure with large dendritic spacing's. Finally, microstructure of the chilled composites is finer than that of un-chilled matrix alloy with uniform distribution of SiO₂ particles. This is due to effective stirring of the composite before pouring and chilling during solidification.

Microstructural characteristics of chilled composites are mainly dependent on reinforcement content in the matrix alloy, their bonding and distribution. In the present research discussion is based on 9 wt.% reinforcement since properties (both mechanical and thermal) enhances up to 9 wt.% (optimum). Further increase in the reinforcement content (to 12 wt.%) it was observed that the cluster formation of the reinforcement takes place that deteriorates the mechanical properties [19]. Microstructural studies conducted on the chilled composites developed containing 9 wt.% reinforcement revealed that there is uniform distribution of the reinforcement without the formation of clusters along with good reinforcement-matrix interfacial integrity (bonding) and significant grain refinement without any micro porosity (**Figure 3**). This is due to difference in density of the reinforcement with the matrix alloy, pre heating of the reinforcement for good wetting properties along with stirring to create vortex. Microstructural studies of the heat treated samples (fig. not shown) also revealed that the matrix is recrystallized completely. Grain refinement of the composites due to heat treatment is primarily attributed to fact that SiO₂ particulates to nucleate in Ni grains during directional solidification that restricted growth of recrystallized Ni grains. Interfacial integrity between matrix and the reinforcement was also assessed using scanning electron microscope for the fractured surfaces (figures not shown) to analyze the integrity of the particulate-matrix interface. Here also SEM studies reveal that a strong bond exists between the interfaces as expected. Therefore, microstructural studies reveal that, faster rate heat transfer during solidification (chilling) of the composite in this research leads to strong bonding of the reinforcement and the matrix and hence the properties. The result of microstructural studies of chilled Ni composites however did not reveal presence of any micro-porosity or shrinkage cavity or there was no evidence of any other defects. This may be one of the main reasons for increase in properties of the composite developed.

3.3. Thermal Analysis

3.3.1. Coefficient of Thermal Expansion (CTE)

The coefficient of thermal expansion (CTE) depicts how much the measure of a material will change with temperature. The magnitude of a given materials CTE is firmly identified with the bond vitality between its constituent atoms.

In the present study the coefficient of thermal expansion is measured for the samples of nickel alloy composite with varying weight percentages of reinforcement (fused silica) cast using copper chill at different temperatures. The main findings from the experiment is that, the coefficient of thermal expansion for

both nickel alloy (base metal) and nickel alloy composite with fused silica reinforcement decreases with the increase in the temperature.

It is observed from the plot (Figure 5) that for the base Ni alloy the CTE varies between 9 to 14 with a maximum at 100°C to a minimum at 250°C and it is noted that CTE for the base Ni-alloy decreases as the temperature increases (from 14 to 9). Figure 5 indicates that for given temperature as the reinforcement content increases the CTE gradually decreases and at 9 wt.% reinforcement, it reaches its minimum value and later increases as reinforcement content increases beyond 9 wt.% reinforcement. This decrease in CTE value with increase in reinforcement content up to 9 wt.% is due to effect of ceramic phase (SiO_2) reinforcement in a ductile (FCC) matrix (Ni). Beyond 9 wt.% reinforcement addition, due to agglomeration and clustering of the reinforcement (as evidenced from the microstructural observation) the CTE value increases. Therefore to obtain least CTE from the automotive application point of view, 9 wt.% addition of reinforcement is optimum. It is also observed for Figure 5 that, irrespective of reinforcement content, highest CTE is observed at the lowest temperature at 100°C and least at 200°C (CTE = 7). Hence it is concluded that reinforcement content in the MMC and temperature has an effect on CTE of the chilled Ni-alloy MMC developed.

One of the noteworthy components of a composite material is the tailorability of its material properties. The coefficient of thermal expansion (CTE) of a composite material is to perform a key part in its application range. It has been realized that a state of micro stress often exists between the phase of the matrix and

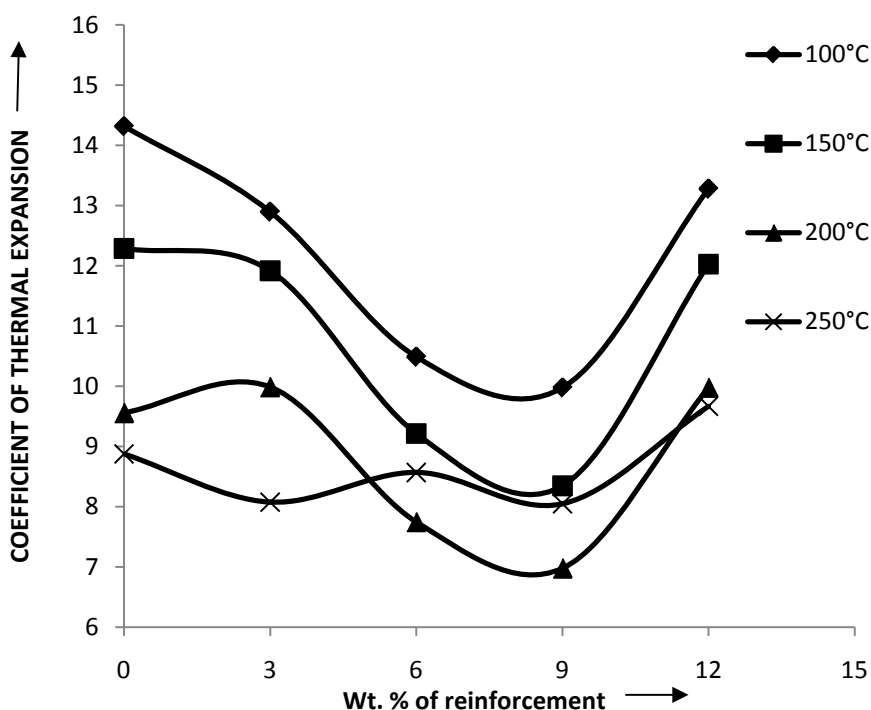


Figure 5. Plot of CTE/s wt.% reinforcement addition for copper chilled nickel alloy at different temperatures.

the reinforcement. Difference in thermal expansion of the individual phases produces stress which indirectly affects the strength properties and modes of failure. The coefficient of thermal expansion of a metal grid composite is difficult to anticipate absolutely in the light of the fact that it is affected by a few elements. For example, the matrix plasticity, the size and state of the reinforcement, the kind of support, the circulation of the reinforcement and the voids in the metal grid composite and so on.

It is observed from the research papers published from other researchers [20] that steel alloyed with Ni and Cr for automotive applications has a CTE value varies between 15 and 22 based on the temperature where as for the present Ni chilled composite, it varies between 7 and 14. Such a low value of CTE of chilled Ni MMC is because of high melting temperature of Ni and SiO₂ reinforcement. Therefore the present chilled Ni MMC is suitable for automotive applications because of its low CTE.

3.3.2. Thermal Conductivity

It is well known that when a material is heated, it expands and the physical measurements change. This is because of the expanded development of the constituent atoms at the elevated temperature. The raise in temperature drives these molecules to keep up a more prominent normal partition separation than they would at a lower temperature. Linear thermal expansion is utilized to focus the rate at which a material expands as a function of temperature. This test can be utilized for design purposes and to figure out whether frustration by thermal stress may happen. Understanding the relative development attributes of two materials in contact can be vital for application achievement.

In the present study of the composite material the thermal conductivity was measured for the specimens of nickel alloy with varying percentages of fused silica cast using copper chill and at 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 decreases continuously with the increase in temperature. The rate of thermal conductivity shrinks with the escalation in weight percentage of SiO₂ was due to the rise of temperature.

It is observed from the **Figure 6** that thermal conductivity for the Ni-alloy without reinforcement at different temperatures varies between 360 w/m.°C to 390 w/m.°C. Highest thermal conductivity observed was 390 w/m.°C tested at 100°C and the least thermal conductivity was 340 w/m.°C at 250°C for chilled Ni MMC containing 12 wt.% reinforcement. From the plot it is observed that with increase the reinforcement content, the thermal conductivity falls continuously irrespective of the temperature. In the case of reinforcement content beyond 9 wt.% and in spite of agglomeration (as evinced from the microstructural observation) of reinforcement, thermal conductivity decreases. A continuous fall in thermal conductivity is attributed to the fact that a ceramic phase *i.e.*, the reinforcement (SiO₂) present in the ductile phase *i.e.*, the matrix (Ni). However it is

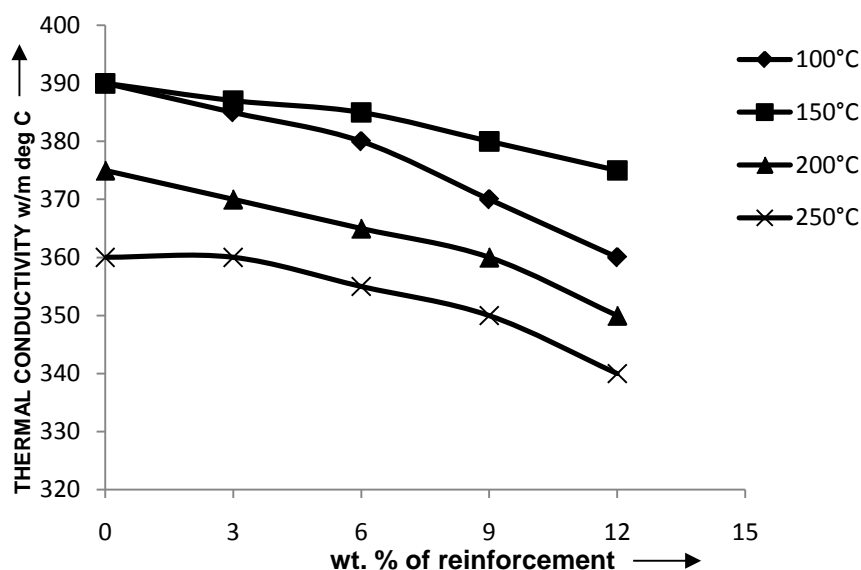


Figure 6. Plot of thermal conductivity V/s wt.% reinforcement for copper chilled Ni-alloy MMCs at different temperatures.

finally concluded that both temperature and reinforcement content has an effect on thermal conductivity.

In this research once again it is observed that, since the thermal conductivity of Ni alloy MMC developed (390 w/m deg K) is more than that of steel (230 w/m deg K), the Ni MMC developed in the present research suits automotive application from the heat transfer point of view to keep the components cool.

3.4. Temperature Distribution (IC Engine Valve) of the Chilled Ni-MMC Developed

It is well known that IC engine exhaust valves at present are made out of Ni-Cr steel alloy which operates at around 650°C. Since the melting temperature of matrix alloy Ni and reinforcement SiO₂ of the present investigation are more than that of steel, it is decided in the present research to replace the existing the steel valve by the chilled Ni-alloy composite developed. Therefore as a practical application of the composite developed, an attempt has been made to study the temperature distribution along the length of the IC engine exhaust valve using Finite Element Analysis (FEA) using the SOLID WORKS station by Microsoft. The properties of the material for FE analysis are given according to the properties of the composite developed (matrix and reinforcement) and boundary condition according to the prevailing IC engine combustion chamber conditions.

Figure 7 shows the standard IC engine exhaust valve which is meshed for FE analysis. **Figures 8-10** shows the temperature distribution along the length of the valve for the base Ni alloy (without reinforcement), Ni alloy with 3 wt.% reinforcement and Ni alloy with 9 wt.% reinforcement. In this paper only 3 and 9 wt.% are considered (data for other additions are available) for discussion because 3 wt.% is the minimum and at 9 wt.% the distribution of the reinforcement

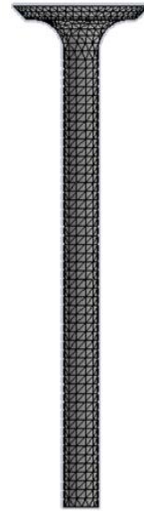


Figure 7. Meshing of IC engine valve.

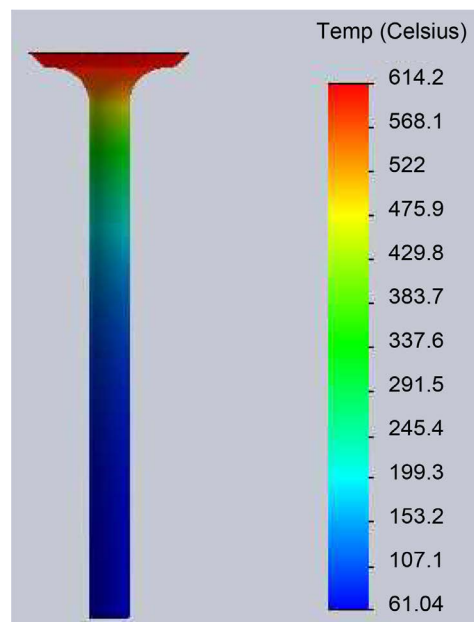


Figure 8. Temperature distribution for base metal (Chilled Ni-alloy).

is uniform in the matrix.

It is observed from **Figures 8-10** that the tip of the valve the temperature is around 63°C and it goes on increases towards the base of the valve. In the case of base Ni-alloy with reinforcement the base temperature was 614.2°C followed by 572°C for 3 wt.% addition of reinforcement and 568.6°C for 9 wt.% addition of the reinforcement. It is also observed from the temperature distribution profile that the temperature decreases monotonically from tip to base and in the case of Ni alloy without reinforcement it was 46°C and for Ni alloy with 3 wt.% and 9 wt.% reinforcement it as 43°C and 42°C respectively. This shows that reinforcement has an effect on the temperature distribution. Again it is observed from the

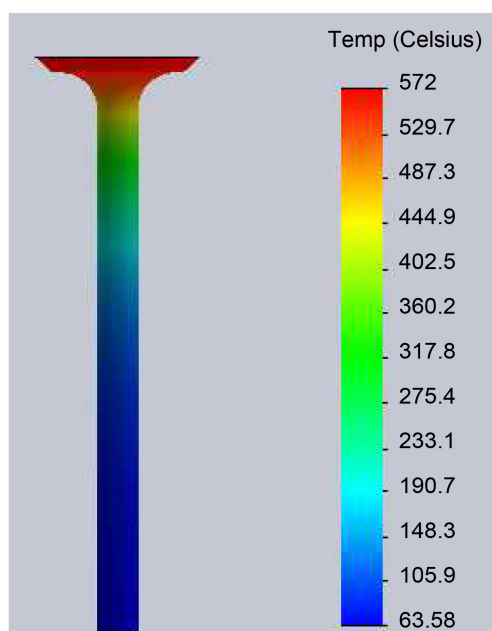


Figure 9. Temperature distribution for chilled Ni-alloy containing 3 wt.% SiO₂.

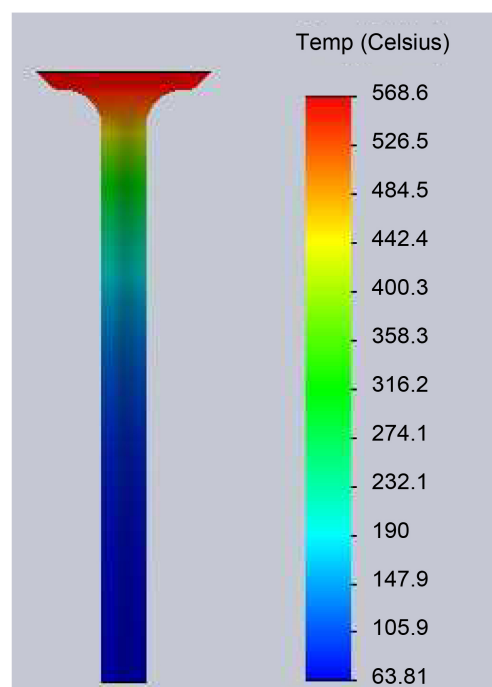


Figure 10. Temperature distribution for chilled Ni-alloy containing 9 wt.% SiO₂.

figures that the highest base temperature (614°C) was recorded for base Ni-alloy without reinforcement and reduces to 572°C for 3 wt.% reinforcement and 568°C for 9 wt.% reinforcement.

Thus it is concluded that Ni alloy MMCs can be conveniently be used as an alternate material for IC engine valves and reinforcement has an effect on the

temperature distribution *i.e.*, base temperature decreases as the reinforcement content increases. In addition to the above, IC engine exhaust valves presently used are made of steel alloy (Ni-Cr) has to work at an operating temperature of 650°C is also subjected to cyclic loading because of their operation and hence they should be tough enough and wear resistant.

But the present chilled Ni MMC developed embedding SiO₂ as the reinforcement possesses all the above properties *i.e.*, Ni alloy and SiO₂ are fatigue and wear resistant. Data obtained regarding temperature distribution and properties of the valve from the standard IC engine valve manufacturers (Escorts and Goetz Ltd. and CAR mobiles Ltd., India) are in consistence with the Ni alloy MMC developed in the present investigation. It is worth to note here that the hardness of Ni is more than that of alloy steel (235 and 216 BHN) and density of Ni is also more than that of steel (8.9 and 7.85 gm/cc) along with a tensile strength of 580 MPa. Hence the present chilled Ni MMCs can be conveniently used for IC engine valves.

4. Conclusions

This research work contributes in providing to develop the chilled ASTM A 494 M grade Nickel alloy composite with varying additions off used SiO₂ starting 3 wt.% towards 12 wt.%. The composites were established through stir cast method via induction heating technique involved with stirring mechanism.

Following are the conclusions of the present research based on the experimental results and the discussions:

1) Copper chilling of the MMCs and the reinforcement content however do fundamentally influence the microstructural and thermal properties of the Ni-alloy MMC developed.

2) The microstructural study reveals that the reinforcement particulates are uniformly distributed in the composite and no interfacial reactions are seen under higher magnifications. It is particularly observed that the chilling effect during solidification has affected the microstructures. Along with this, the addition of SiO₂ has affected the morphology of the nickel alloy. Microstructure of the chilled composites is finer than that of un-chilled matrix alloy with uniform distribution of SiO₂ particles. Strong interfacial bond was observed with no agglomeration between the matrix and the reinforcement (up to 9 wt.% of the addition). This is due to effective stirring of the composite before pouring.

3) A decrease in CTE value was observed with increase in reinforcement content up to 9 wt.% and is due to effect of ceramic phase (SiO₂) reinforcement in a ductile (FCC) matrix (Ni). It is also observed that, irrespective of reinforcement content, highest CTE is observed at the highest temperature at 250°C and least at 100°C. Hence it is concluded that reinforcement content in the MMC and temperature has an effect on CTE of the chilled Ni-alloy MMC developed.

4) It is observed that thermal conductivity for the Ni-alloy without reinforcement at different temperatures varies from 360 W/m·°C to 390 W/m·°C. Highest

thermal conductivity observed was $390 \text{ W/m}^\circ\text{C}$ tested at 100°C and the least thermal conductivity was $340 \text{ W/m}^\circ\text{C}$ at 250°C for chilled Ni MMC containing 12 wt.% reinforcement. Hence both temperature and reinforcement content has an effect on thermal conductivity.

5) FE analysis showed that reinforcement has an effect on the temperature distribution. Again it is observed from the figures that the highest base temperature (614°C) was recorded for base Ni-alloy without reinforcement and reduces to 572°C for 3 wt.% reinforcement and 568°C for 9 wt.% reinforcement. Thus it is concluded that Ni alloy MMCs can be conveniently be used as an alternate material for IC engine valves and reinforcement has an effect on the temperature distribution *i.e.*, base temperature decreases as the reinforcement content increases.

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