Effect of Deep Cryogenic Treatment on Microstructure, Mechanical and Fracture Properties of Aluminium-Al_2o_3 Metal Matrix Composites

Panchakshari H.V., Girish D. P., M Krishn

Abstract: ***The aim of this research work was to focus on the effect of deep cryogenic treatment on the microstructure, mechanical and fracture properties of Al6061/Al_2O_3 metal matrix composites (MMCs) at -196 °C for different time duration. Al/Al_2O_3 metal matrix composites containing 5, 10, 15 and 20% of Al_2O_3 are produced by liquid metallurgy technique. After deep cryogenic treatment of samples at liquid nitrogen temperature, the microstructure of specimens shows the change in distribution of precipitates. The precipitate particles almost dissolved in the matrix and obtained very fine grain boundaries. The modification of microstructure of MMCs due to cryogenic treatment shows significant improvement in mechanical properties of the MMCs. The preferred orientation of grains was sufficiently corroborated by XRD results of Al/Al_2O_3 composite before and after cryogenic treatment.***

Keywords: Metal Matrix Composites, Cryogenic treatment, microstructure, microhardness

I. INTRODUCTION

Cryogenically treated nonferrous metals will exhibit longer wear and more durability. This growing field of engineering is an excellent technique to improve the mechanical properties of metals [1]. Cryogenic treatment enhances metallurgical properties of most of the metals, which in turn improve various strengths of the treated parts [2]. Cryogenic treatment creates denser molecular structure of the metals and alloys resulting in a larger contact surface area that reduces friction, heat and wear. The effect of cryogenic cooling on the tensile properties of metal-matrix composites (MMCs) has been studied by Poza [3]. The beneficial effects of the cryogenic cooling resulted from an alteration of the residual stress state brought about by plastic flow of the MMCs. Consolidated MMCs showed exceptionally high hardness values, which are attributed to the formation of ternary phases at the temperatures of consolidation. The samples consolidated at high temperature possessed low fracture toughness which is also attributed to the formation of ternary phases during consolidation. Hemanth [4] prepared B_4Cp/Al-12%Si composites in which Al-12% Si alloy was the matrix and boron carbide particles (B_4Cp) were the reinforcing material. The characterization of tribological behavior of cryo-chilled Al-B_4C composites castings with high rate heat transfer during solidification was studied. He observed that, the microstructures of chilled composites are finer than that of the un-chilled matrix alloy with uniform distribution of B_4C particles. Although few researchers [3-5] were working on effect of cryogenic treatment on mechanical properties of Al MMCs but no published research work focused on the effect of cryogenic treatment on microstructural changes and fracture behavior of MMCs. These parameters are important to study the behavior of composites after cryogenic treatment. Hence this investigation was focused on effect of cryogenic treatment on microstructure, mechanical and fracture properties of Al/Al_2O_3 MMCs.

II. EXPERIMENTAL PROCEDURE

The Al 6061 alloy (matrix material) and Al_2O_3 30-50 μm size particles (reinforcement) were used for fabrication of Al6061/Al_2O_3 MMCs. The chemical composition of Al6061 is given in the Table 1. The reinforcement particles were chosen as commercial Al_2O_3 with 99.5% purity.

The Al_2O_3 of 30-50 μm size were used as the reinforcement and the Al_2O_3 content in the composites was varied from 5 to 20% in steps of 5% by weight. Liquid metallurgy technique was used to fabricate the composite materials in which the Al_2O_3 particles were introduced into the molten metal pool through a vortex created in the melt by the use of an alumina-coated stainless steel stirrer. The coating of alumina on the stirrer is essential to prevent the migration of ferrous ions from the stirrer material into the molten metal. The stirrer was rotated at 550 rpm and the depth of immersion of the stirrer was about two-thirds the depth of the molten metal. The pre-heated (773 K) Al_2O_3 particles were added into the vortex of the liquid melt which was degassed using pure nitrogen for about 3 to 4 min. The resulting mixture was tilt poured into preheated permanent moulds

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TABLE 1 CHEMICAL COMPOSITION OF AL 6061

Mg	0.92
Si	0.76
Fe	0.28
Cu	0.22
Ti	0.10
Cr	0.07
Zn	0.06
Mn	0.04
Be	0.003
V	0.01
Al	Bal

Cryogenic treatment of samples has been performed by placing Al and Al/Al_2O_3 specimens in an isolated alumina chamber. The top of the chamber was covered by insulator glass wool after placing the samples in the chamber. This chamber was progressively immersed in a liquid nitrogen reservoir. The sample temperature was monitored by a K type thermocouple which was used to operate a stepper motor to lower the position of samples and maintained a temperature decline at the rate of 1 °C/min. It took about 4 hours to reach − 196^0C. This method eliminates the probability of thermal shock and micro-cracking. Specimens were held at − 196^0 C for 10, 20, 30, 40 and 50 h and then slowly brought up to approximately + 25 °C. The cryogenic procedure is followed as per Kaveh Meshinchi et.al. [6]. the specimens for optical microscopy were prepared according to ASTM E3 standards. The samples were first subjected to grinding and polishing followed by etching by nital. Optical micrographs were taken using the Olympus metallurgical microscope, fitted with a camera. Specimens were washed with distilled water followed by acetone and dried thoroughly.

All mechanical tests were conducted in accordance with ASTM standards. Tensile tests were conducted at room temperature using universal testing machine (UTM) in accordance with ASTM E8-82. The tensile specimens of diameter 8.9 mm and gauge length 76 mm were machined from the cast composites with the gauge length of the specimen parallel to the longitudinal axis of the castings. Five specimens were tested and average values of the UTS and ductility were measured. Fracture surface was studied using scanning electron microscope. The compression tests were conducted on the specimen with12 mm diameter and 20 mm length machined from the cast composites. In these tests, the compressive loads were applied gradually and corresponding strain were measured until failure of the specimen occurred. This test was conducted according to ASTM E9 standard on the UTM at room temperature.

III. RESULTS AND DISCUSSION

3.1 Microstructural investigation

The micrograph of the specimens before and after cryogenic treatment of both Al matrix and Al/20%Al_2O_3 MMCs are given in the Fig.1. Being able to measure the discrepancy of the region of interests before and after cryogenic treatment as shown in the images would be a strong determinate to support the assertion of increased mechanical properties. Figs. 1(a), (b), (c) and (d) show optical microscopy images of the microstructure of as cast Al, as cast Al /20 wt. % Al_2O_3 MMCs, 50h cryogenic treated Al matrix alloy and 50 h cryogenic treated Al/20 wt.% Al_2O_3 MMCs respectively. The execution of cryogenic treatment had a significant effect on the microstructure of the MMCs and led to transformation of α-Al to β ($Mg_{17}Al_{12}$) phase. In the as-cast Al alloy, the β phase exhibited irregular morphologies (eutectic β phase) and tiny laminar shaped morphologies it is confirmed in the XRD Fig. 2(b). It is also confirmed by Mehta et al. [7]. They proved that, the β phase has the main strengthening effect on the Al matrix alloys at room temperature. Kaveh Meshinchi et. al [8] showed that cryogenic treated Al alloy has little higher melting temperature and is the main reason for higher mechanical properties at room and elevated temperatures. The coarse divorced β phase penetrated the matrix alloy as confirmed by TEM studies. As a result of this change in the microstructure, the grain size of cryogenic treated samples decreased when compared with the as-cast specimen as shown in Fig.1. This improvement was attributed to the strengthening of the matrix against propagation of the existing defect which is due to the important role of β precipitates in the microstructure which are the main strengthening effect at room temperature.

The XRD crystallograms before and after cryogenic treatment of these alloys were studied and as shown in Fig. 2. It reveals that the cryogenic treatment can change the diffraction peak intensity of some crystal planes in these alloys. Fig. 2(a) shows XRD patterns of Al /Al_2O_3 MMCs before cryogenic heat treatment, the range of incident angle is between 30°and 100°. The target is Cu Ka, the tube voltage is 40kV and the electric current is 60 mA. The properties of MMCs are related to its XRD patterns gained from the surface of MMCs.

Fig.2(b) shows the XRD patterns for virgin surface of Al MMCs. The XRD patterns after cryogenic treatment shows all the peaks are consistent except for the plane (111). The width of the peak decreased to half. This indicated that the grain in MMCs become small after cryogenic treatment. The studying of Govindan Potti et al. [9] shows that the crystallization strengthens after the specimens have been cryogenic treated as shown in TEM Fig 3

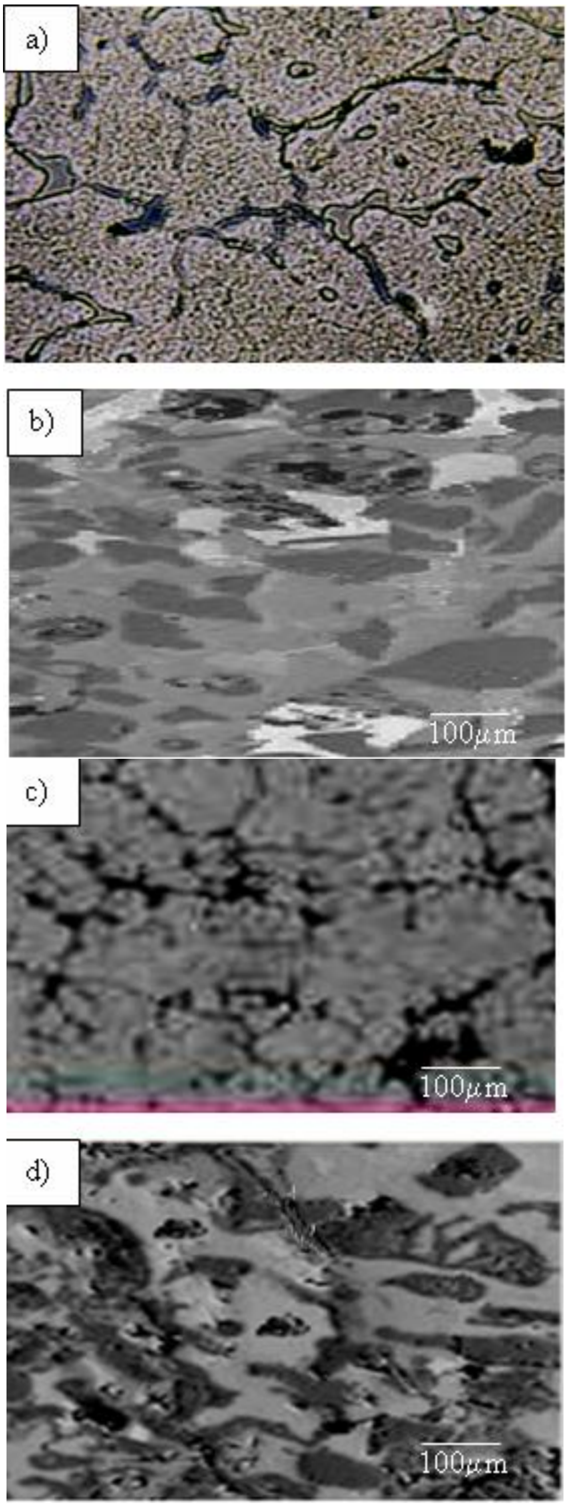


Fig.1. Optical micrographs to show the surface of as cast specimens of a) Al matrix alloy and b) Al/20% Al_2O_3 MMCs. c) Al matrix alloy -50 h cryogenically treated. d) Al/20% Al_2O_3 MMCs -50 h cryogenically treated

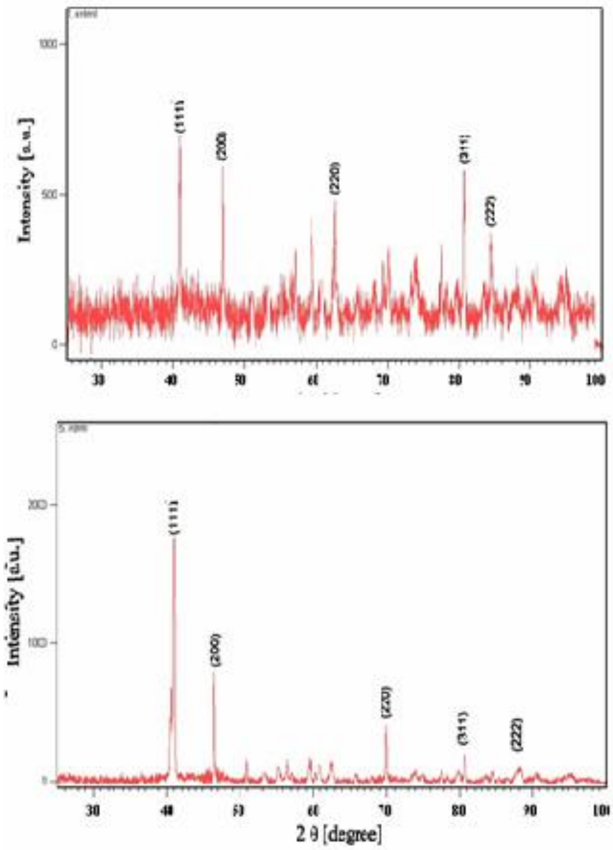
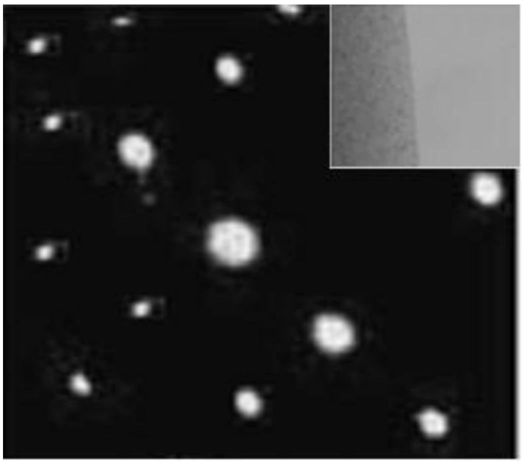


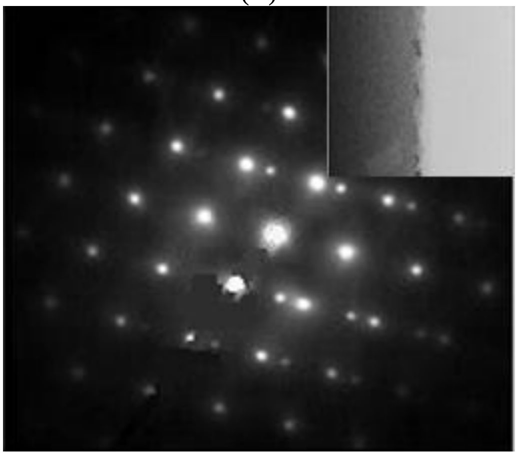
Fig.2. XRD patterns of Al/Al_2O_3 MMCs before and after cryotreatment

Fig.2(b) shows the XRD patterns for virgin surface of Al MMCs. The XRD patterns after cryogenic treatment shows all the peaks are consistent except for the plane (111). The width of the peak decreased to half. This indicated that the grain in MMCs become small after cryogenic treatment. The studying of Govindan Potti et al. [9] shows that the crystallization strengthens after the specimens have been cryogenic treated as shown in TEM Fig 3

In the as-cast condition, no reaction layer between reinforcement and matrix is visible as shown in right top position of Fig.3(a) whereas in the cryotreated Al/Al_2O_3 MMCs an interfacial reaction layer inhomogeneously surrounding the particle was found in the right top view of Fig. 3 (b). The reaction products have been identified by electron beam diffraction as Mg_2Si and MgO.



(A)



(B)

Fig.3 TEM images of Al / Al_2O_3 MMCs. A) as cast condition B) after cryogenic treatment with diffraction pattern of reaction products.

3.2 Mechanical properties

The results of the mechanical tests such as ultimate tensile strength, yield strength, hardness, compression strength and ductility of both as cast and cryogenic treated Al and Al/Al_2O_3 MMCs are given in the Fig. 4, Fig. 5, Fig. 6, Fig. 7 and Fig. 8 respectively.

3.2.1 Ultimate tensile strength

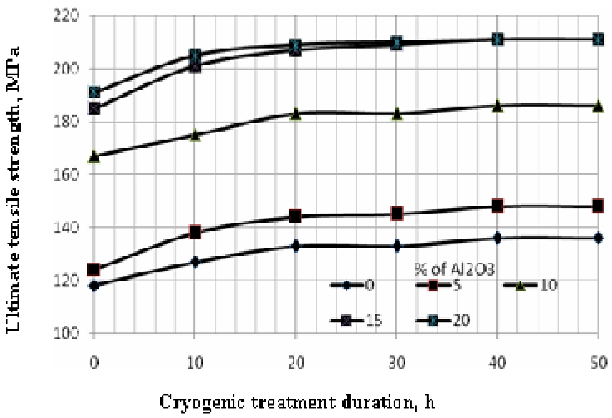


Fig. 4 Effect of cryogenic treatment duration on ultimate tensile strength of Al and Al/Al_2O_3 MMCs

From the Fig. 4 it is obvious that, the Al/Al_2O_3 MMCs exhibits higher ultimate tensile strength (UTS) than the Al matrix materials. Consequently the UTS are not only dominated by the particles, but also influenced by the properties of matrix and particle interface. Cryogenic treatment seems to increase the UTS monotonically by significant amounts for the composites of various Al_2O_3 contents. The maximum percentage increase in UTS of the composites is 16%. Cryogenic treatment causes the matrix to be hardened and it removes internally locked residual stresses, allowing easier movement of dislocations. UTS have been increased upto 30 hours treatment, the further cryotreatement is not been much effective and in some cases there is a little decline in the UTS due to saturation. Also it is observed from the figure that, at higher percentages of Al_2O_3, the formation of interfacial reaction products (MgO and Mg_2Si) during cryogenic treatment increases the composite strength significantly. High cooling rate has caused the formation of small dendrites which enhances the strength of the materials. In the previous work, it was proved that, although the β phase has the main strengthening effect on the Al-Mg based alloys at room temperature; it has a low melting temperature and is the main reason for poor mechanical properties of these alloys at elevated temperatures [14].

3.2.2. Yield strength

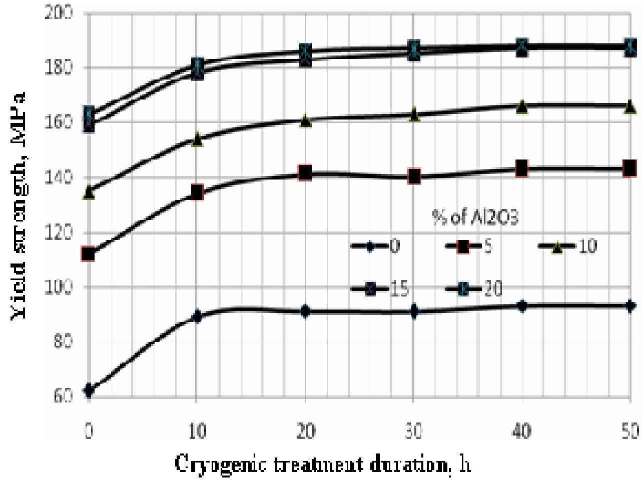


Fig. 5 Effect of cryogenic treatment duration on yield strength of Al and Al/Al_2O_3 MMCs

Fig.5 shows the yield strength of Al and Al/Al_2O_3 composites with varying compositions of Al_2O_3 and different cryogenic treatment duration. It may be inferred from the graph that, as the percentage of Al_2O_3 particulates increases from 0 to 20% by weight, there is increasing in yield strength from 60 to 160 MPa for as cast condition and up to 185 MPa for cryotreated condition. Kennedy & Wyatt[10], obtained similar results in particulate reinforced Al composites and observed an increase in yield strength with addition of particulates regardless of the type of reinforcement used. It can be seen that, the improvement of yield strength is up to 30 hours of cryogenic treatment and further very little improvement was observed. The cryogenic treatment allows diffusion of segregated components, producing a more uniform composition which has enhanced the yield strength of both matrix alloy and composites.

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3.2.3 Hardness

Fig. 6 shows the effect of increase in particulate content and cryogenic treatment duration on the hardness of Al/Al_2O_3 MMCs. The increase in hardness was about 43% as Al_2O_3 particulate was increased from 0 to 20 wt%. The increased hardness is attributed to the presence of hard ceramic particles, which act as barrier to the movement of the dislocation with the matrix. The cryogenic treatment increases the hardness around 10 -15% both matrix material and composites.

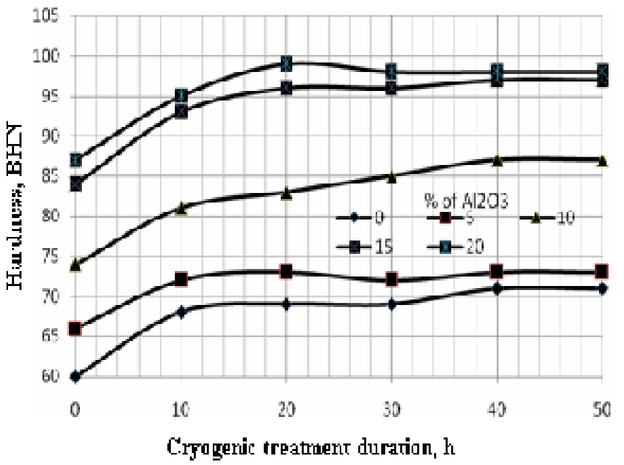


Fig. 6 Effect of cryogenic treatment duration on hardness of Al and Al/Al_2O_3 MMCs

The hardness of the both alloy and composites is influenced by the dislocation movement in the substructure due to cryogenic treatment. The higher vacancy concentration is obtained by lowering the temperature. This promotes the clustering process and results in a finer scale of precipitation.

3.2.4 Compression strength

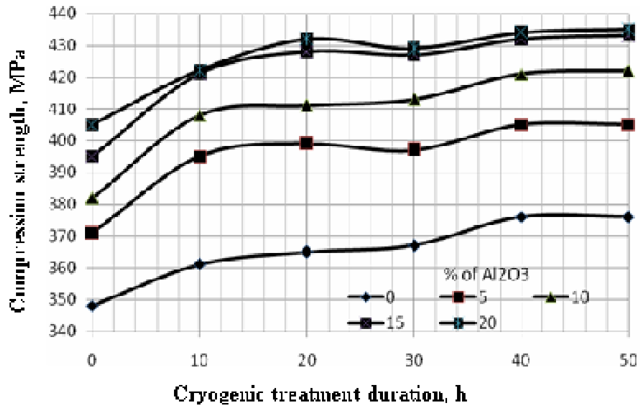


Fig. 7 Effect of cryogenic treatment duration on ultimate compression strength of Al and Al/Al_2O_3 MMCs

As shown in Fig. 7, the compression strength of the Al and Al/Al_2O_3 MMC increases with the addition of Al_2O_3 particulate percentage and duration of cryogenic treatment. The compressive strength of as-cast composite material increased significantly by 18% as the Al_2O_3 particulate content was increased from 0 to 20 wt.% if other factors were kept constant. Seah et. al [11] compared the compression and tensile properties of lead base MMCs and observed similar results. The increase in compression strength is attributed to secondary hard ceramic dispersoids, which increases dislocation density. Due to hard ceramic dispersoids, the MMCs behave as brittle material rather than ductile material, which were evident from the results, obtained. Dutta & Surappa[12] explains that, the reinforcement causes a high dislocation density in the matrix, resulting in improved compressive strength. Cryogenic treatment increased the compressive strength by significant amounts (upto 10%). The results are on par with those obtained by Jiang et. al [13] who found that the cryo-treatment resulted in the microstructure becoming more homogeneous, causing the compressive strength to improve.

3.2.5 Ductility

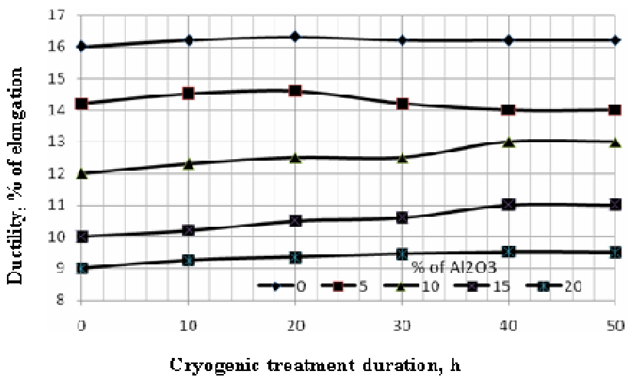
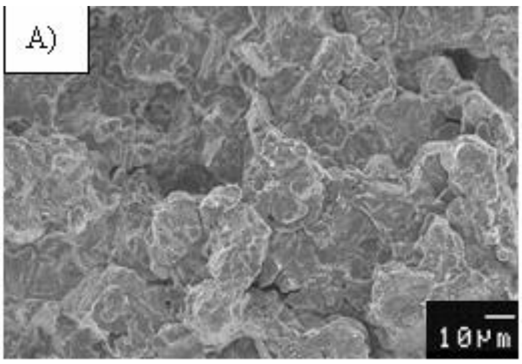


Fig. 8 Effect of cryogenic treatment duration on ductility of Al and Al/Al_2O_3 MMCs

The ductility is measured in terms of percentage elongation. The percentage elongation measured for ascast and cryogenic treated composites are shown in the Fig.8. The ductility decreases with the increase in Al_2O_3 content by a significant amount. The reduction in ductility was about 53 % as the Al_2O_3 content was increased from 0 to 20 wt%. Olivier et al.[14] are of the opinion that this behavior is probably due to void nucleation during plastic straining of the reinforcement, either by reinforcement interface or by the de-cohesion of the matrix-reinforcement interface. Some authors believe that, the compressive-to-tensile stress transition for a critical volume fraction could be described as one of the contributing factors for the steep decrease of ductility and fracture toughness of the composites with increasing volume fraction



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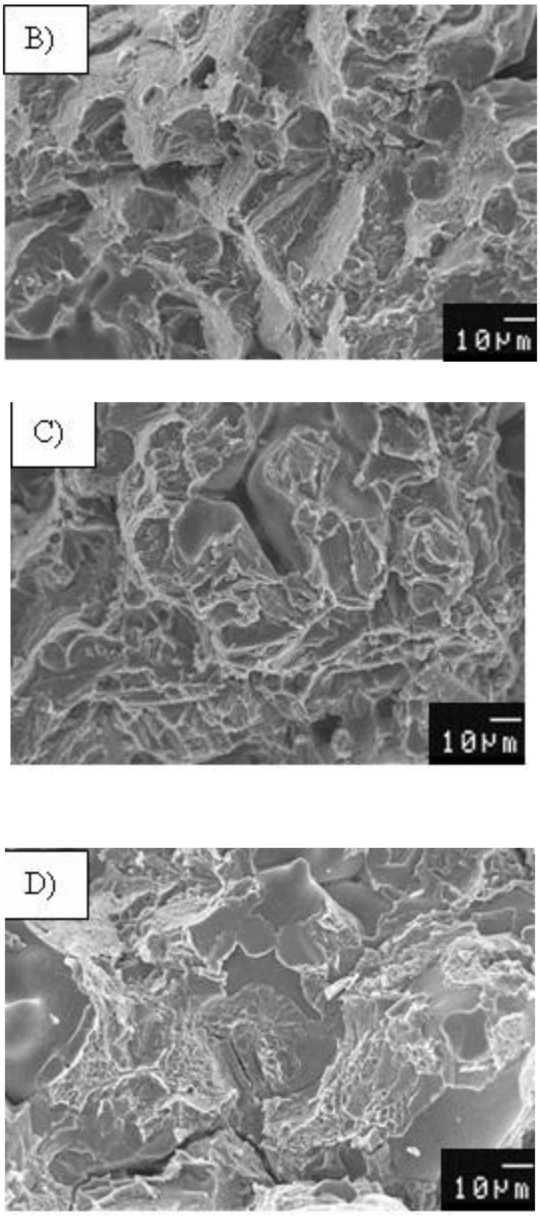

Fig.9. SEM fractographs of the a) as cast Al matrix alloy, b) as cast Al/Al_2O_3 MMCs, c) cryogenic treated Al matrix alloy and d) cryogenic treated Al/Al_2O_3 MMCs.

The ductile failure in the matrix caused by the nucleation, growth and coalescence of voids from the cracking of the intermetallic inclusions and dispersoids in the matrix, by ductile tearing of the matrix between the reinforcement [15]. Cryogenic treatment was found to slightly improve the ductility of the composites. A variation in density of MMCs can be attributed to the annihilation factor of lattice defect, the cryogenic treatment results in improved homogeneous nature of composites, which increase the ductility of MMCs [16]. By cryogenic treatment, there is a possibility of plastic yielding which relieves residual stresses in the composite specimens, reducing mismatch strain which may be one of the reasons for the increase in ductility of both alloy and MMCs.

Fracture Behavior

In the as cast condition, both Al alloy and the composites experienced ductile type fracture behaviour. Primary dimples originated from the reinforcement particles. Whereas micro-voids of smaller size grew from matrix finer precipitates. The systematic analyses of the broken specimens showed that, the fracture surface were generally broken as shown in Fig. 9.both in ascast and cryotreated condition. Micro-voids were thus considered to form due to reinforcement fracture under the action of load. The unbroken particles of cryo-treated specimens were found on the fracture surface. Fragment of interface reaction product were seen. Particle-matrix interface de-bonding thus acted as a preferential mechanism of fracture nucleation.

Al alloy and Al MMCs for cryogenic treatment are qualitative to the shear band pattern, which determines the homogeneity of deformation. The ductility is largest when only 45^0 matrix shear bands appear (ascast condition). In materials that exhibit long-reaching damage-induced shear bands, the fracture strain depends on the sizes of the areas containing matrix shear bands. Particle clusters cause inhomogeneous local deformation behavior, similar to MMCs with pre-damaged large particles. Most brittle are those materials, where damage-induced deformation bands appear at 90^0.

IV. CONCLUSIONS

1. Cryogenic treatment changes the morphology of precipitate in both Al alloy and Al/Al_2O_3 MMCs. The coarse eutectic β phase present in the matrix material in MMCs resulted in the improvement of hardness of cryogenic treated samples compared with as-cast samples.
2. Important microstructural changes occur during the initial cryogenic treatment examination. The significant changes were noted in the diffraction patterns with increased duration of cryo-soaking. At the beginning of cryotreatment, very small solute clusters were formed. Which is evident from the fluctuations in concentration with periodicity along <111> direction in the matrix.
3. The mechanical properties such as UTS, yield strength, compression strength and hardness increase with increasing wt. % of reinforcement for the MMCs for the same cryogenic condition. The application of cryo treatment has increased the hardness further.
4. The fracture surface of cryogenic treated Al and Al MMCs is qualitative to shear band pattern which determines the homogeneity of deformation compare to ascast condition.

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