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Enhancement in Microhardness and Microstructural changes through Cryogenic treatment of LM25+TiO2 Metal matrix composite

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Abstract: *The aim of this experimental investigation is to increase awareness amongst the researchers and to draw their attention towards the present approach to deal with the cryogenic treatment for the nonferrous metals. Cryo means cold born crystal structure in metallurgical aspect. Cryogenic treated nonferrous metals will exhibit longer wear and more durability. During metal making process, when solidification takes place, some molecules get caught in a random pattern. The molecules do move about at subzero and deep cryogenic treatment slowly. In this experimental study the effect of cryogenic treatment on microstructure changes and the hardness properties varies for LM25 alloy and LM25-TiO2 metal matrix composite at -196°C.It is analyzed for different durations. The execution of cryogenic treatment on both alloy and MMCs changed the distribution of β precipitates. The XRD crystallogram reveals that the cryogenic treatment can change the diffraction peak intensity of some crystal planes in MMCs. The influences of different volume fraction of reinforcement and cryogenic process parameters on microhardness of LM25 alloy and composite were compared with alloy and composite without cryo treatment and the results showed that the cryogenic treatment improves the hardness of LM25/TiO_2 composites.*

Keywords: *MMCs, Cryogenic treatment, Microstructure, Micro hardness*

I. Introduction

The enhanced performance of the MMCs are seen in the wide range of application starting from toys to high performance requiring areas like automobile industry and aerospace industry due to their weight saving characteristics and providing high beneficiaries over existing materials[1]. To further increase the properties of these composites several process like heat treatment for whole material and surface processing is also being carried out, In other hand cryogenic treatment also known as subzero treatment is done which is very old process and widely used for the high precision parts and objects especially for the ferrous materials earlier [2]. The subjecting of the material to extreme cold hardens and strengthens the material is used long time ago for centuries [3]. Now the cryogenic treatment is widely used in many industries automotive, aerospace, electronic and mechanical engineering industries to improve mechanical strength and dimensional stability of components [4]. For the past few years the cryogenic treatment for the nonferrous metals such as aluminium and magnesium alloys has been done for the their improvement of properties[5]. The mechanical properties and microstructure of the metals and alloys in cryogenic treatment drew the attention of researchers towards this process. The researchers [6,7] showed the beneficial effects of cryogenic treatment on nonferrous metal aluminium. The effect of cryogenic treatment on the wear performance of copper alloy showed least significant changes [8]. The researcher Adam [9] showed the significant improvement of the mechanical properties strength, hardness, toughness of aluminium alloy when subjecting it to cryogenic treatment. This lead to the idea of analyzing the properties when a MMCs is undergoing cryogenic treatment. This field is rapidly growing and is used by many manufacturers. The present work intends to construct a facility to research the process and results of the cryogenic treatment. This helps to create standards for both processing and testing that are currently unavailable. Hence it gives importance that mechanical properties of the MMCs developed are evaluated at cryogenic temperatures. Thus in this experimental work cryogenic treatment was applied to LM25/TiO_2 MMCs to study its effect on microstructure and hardness of LM25/TiO_2 MMCs.

II. Experimentation

The metal matrix composite is prepared by stir casting method by taking matrix material as LM25 alloy and the particulate reinforcement as TiO_2 upto a volume fraction of 20%. The chemical composition of LM25 alloy is given in the table 1. The reinforcement particles were chosen as commercial TiO_2 with 99.5% purity.

Table 1 Chemical composition of LM25 aluminium alloy

Cu	Si	Mg	Mn	Fe	Ti	Ni	Zn	Sn	Al
0.17	**6.81**	**0.51**	**0.03**	**0.29**	**0.103**	**0.006**	**0.06**	**<0.001**	**balance**

The TiO_2 of 30-50 µm size were used as the particle reinforcement in the composite material. The reinforcement particle is varied in volume fraction by 5 to 20 % to get different composites. The reinforcement particle is preheated to a temperature of 550°C to remove the moisture content before adding into the molten aluminium.

The liquid metallurgy technique is used to fabricate the composite, in which the preheated TiO_2 particle is introduced into the molten pool in the vortex created in the melt, by the use of power operated stirrer, its speed is maintained as 550 rpm and the stirrer is coated with alumina to prevent the migration of ferrous ions from the stirrer material to the molten metal. The depth of immersion of stirrer was two thirds the depth of the molten metal. The resulting mixture of LM25 alloy and TiO_2 is tilt poured into the preheated permanent mould. Thus the various volume fraction of reinforced SiC composite material is fabricated[10].

Cryogenic treatment of samples is performed by placing LM25 alloy and LM25/TiO_2 specimen in a cryogenic chamber. This chamber is progressively immersed in the liquid nitrogen reservoir. The sample temperature is monitored by a K type thermocouple which was used to operate a stepper motor which lowered the sample and maintained a temperature decline at the rate of 1°C/min. The temperature is lowered to -196°C the time taken to reach is nearly 4 hours.

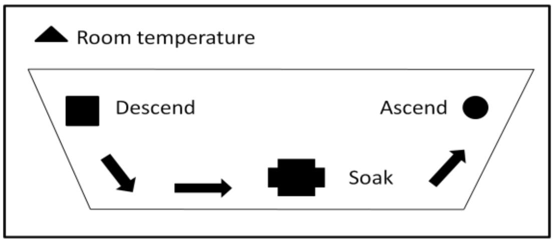
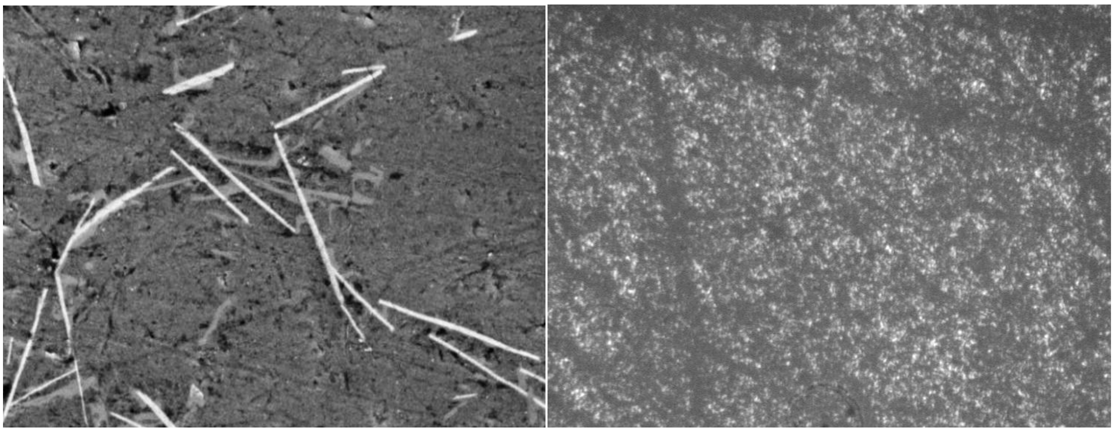


Fig 1 Cryogenic processing

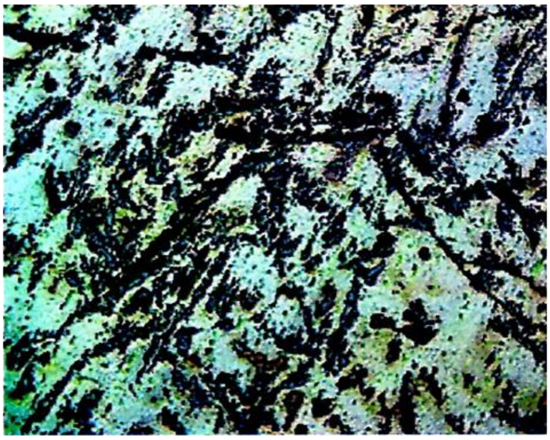
The pain stacking method is very slow microprocessor controlled process which eliminates the probability of thermal shock and micro-cracking. The sequence of process is shown in Fig 1.Specimen were held at -196°C for various time duration such as 1,5, 10, 20, 30, 40, 50 hours and slowly brought up to approximately +25°C. The cryogenic processing is followed as per Kaveh Meshinchi et al [5]. After the completion of cryogenic processing the specimen is prepared for microstructure analyzes according to ASTM E3 standards. The samples were subjected grinding and polishing followed by etching by nital. The optical microscope was taken using metallurgical microscope and then the specimen is washed with acetone and dried thoroughly for the hardness test. The micro hardness test was conducted using a Leitz Wetzler (Germany) microhardness tester equipped with a Vickers diamond pyramid indenter. The load applied was 1N.

III. Results And Discussion



(a) **(b)**

Fig 2. Microstructure of (a) LM25 alloy and (b) LM25+TiO_2 MMCs before cryogenic treatment



(a)

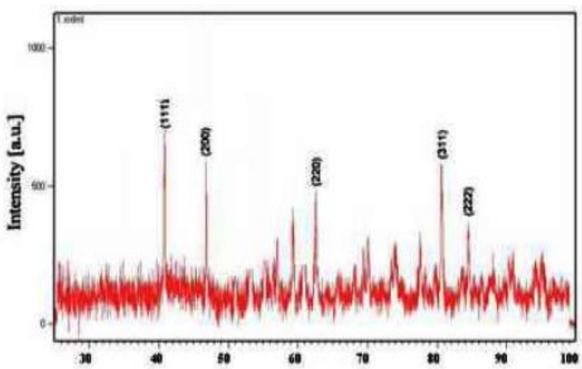
(b)

Fig 3. Microstructure of (a) LM25 alloy and (b) LM25+TiO_2 MMCs after cryogenic treatment

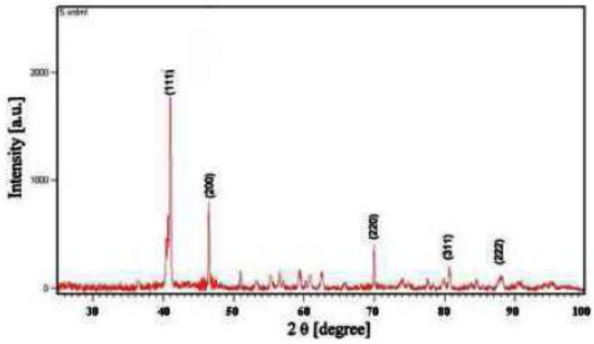
Fig 2.and Fig 3. Shows the microstructure image of the LM25 alloy and its composite before and after cryogenic processing with 10% volume fraction of TiO_2. The impact of cryogenic processing has significant changes in the microstructure of MMCs and led to the transformation of α-Al to β ($Mg_{17}Al_{12}$) phase. In the LM25 alloy the β phase exhibited irregular morphologies (eutectic β phase) and tiny laminar shaped morphologies. The β phase has main strengthening effect on Al-Mg based alloys at room temperature proved by Mehta et al. [11]. The lower mechanical properties at elevated temperature is due to the low melting point of these alloy Kaveh meshinchi et al. [5].

The cryogenic treatment of MMCs of LM25+TiO_2 lead to the changes in microstructure as shown in Fig 2 (b). The coarse divorced β phase penetrated the matrix alloy.

This improved hardness is 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.



(a)



(b)

Fig 4. (a) and (b) XRD patterns of LM25+TiO_2 MMCs before and after cryotreatment

The XRD pattern before and after cryogenic treatment was studied. It reveals that the cryogenic treatment can change the diffraction peak intensity of the crystal planes in these alloys. Fig 4(a) shows the XRD pattern of LM25+TiO_2 MMCs before cryogenic 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.

In fig 4. (b) for virgin surfaces of Al MMCs, it is mainly consistent with the standard pattern for FCC Al. The XRD pattern after cryogenic treatment, all the peaks are consistent but for half width of (111) peak decrease. This indicates that grain in MMCs becomes large after cryogenic treatment. The researcher Govindan potti et al. [12] showed that the crystallization strengthens after the specimen have been cryotreated. Due to this change in microstructure the hardness of the cryogenic treated samples increased compared with the as cast specimen with no treatment as shown in fig 5 and fig 6.

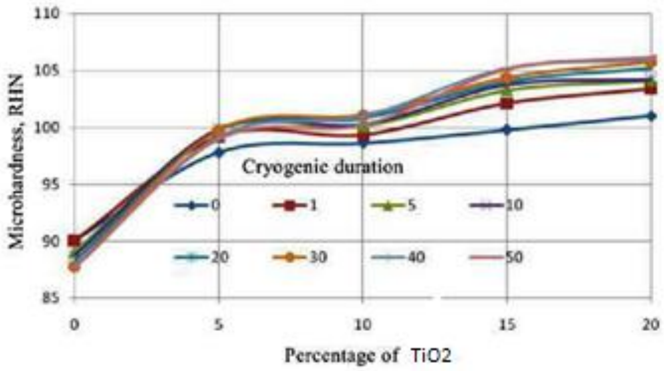


Fig 5. Effect of reinforcement on hardness of the LM25+TiO_2 MMCs

The fig 5. shows that percentage of reinforcement and cryogenic duration on hardness properties of MMCs respectively. It clearly depicts the increase in hardness for the increase in reinforcement percentage and the cryogenic duration in hours.

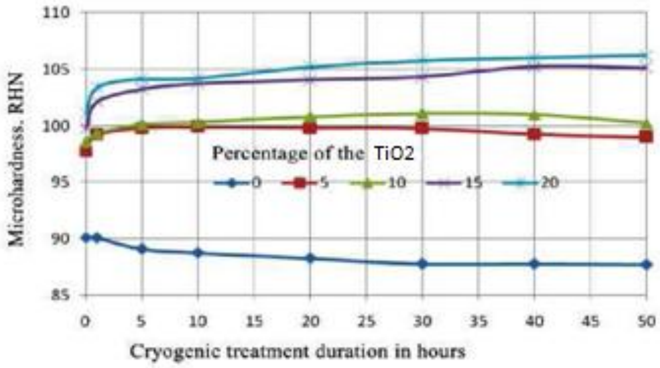


Fig 6. Effect of cryogenic treatment duration on microhardness of LM25 alloy and LM25+TiO_2 MMCs

The LM25 alloy the result shows the hardness decreases with increasing cryogenic treatment. The strength improvement was attributed to the strengthening of the matrix against propagation of the existing of existing defects which is due to the important role of β precipitates in the microstructure which are main strengthening effect at room temperature. Since the β precipitates are mainly distributed at the grain boundaries, the morphology of the β particles after cryogenic treatment and the stabilization of internal microstructure. In LM25 alloy discontinues precipitation of β dominant at grain boundaries, weakens the grain boundaries and hence reduce the hardness with increasing cryogenic duration. The XRD observation showed the cryogenic treatment caused the grain boundaries to broken as a result many grains with the size of 1-3µm. The broken equi axial grains MMCs improve the hardness of the matrix alloy

IV. Conclusion

1. Cryogenic treatment changes the morphology of precipitates in both LM25 alloy and the LM25+TiO_2 composite. The hardness of the cryogenic treated MMCs samples improves due coarse eutectic β phase present in the matrix compared with as cast samples.
2. The hardness increase with the increasing volume percentage of reinforcement for the MMCs for the same cryogenic condition. The application of cryo treatment has increased the effects of particulates with the increase in particulate percentage but pure Al decreased the effect of Cryo treatment.
3. Microstructural change occur during initial cryogenic treatment which is felt in the changes in the diffraction pattern in XRD.

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