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# Investigation on Effect of Cryogenic Parameters on Wear Behaviour of Aluminium-$AL_2O_3$ MMCSS using Taguchi Method

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***Abstract: The objective of this research work was to investigate the effect of cryogenic parameters such as cryogenic temperature (-100, -150 and -196°C), duration of cryogenic treatment (0, 25 and 50 h) and wt. % of reinforcement (0, 10 and 20 wt. %) on wear behavior of Al/$Al_2O_3$ metal matrix composites (MMCs). The $Al_2O_3$ particulate reinforced MMCs were fabricated by liquid metallurgy technique. Specimens so prepared were treated for different cryogenic temperatures for different treatment duration using liquid nitrogen. Both un treated and cryogenic treated specimens were tested on pin-on-disc for constant load 20 N, sliding speed of 1.5 m/s and sliding distance of 3 km.***

***Taguchi method is applied for predicting the optimum cryogenic treatment parameter that gives the lowest wear rate to the castings. The experimental and analytical results showed that the Taguchi method was successful in predicting cryogenic parameters that give the lowest wear rate and the wt. %.***

***Keywords: MMCs, Cryogenic treatment, microstructure, Wear rate, Taguchi.***

## I. INTRODUCTION

Modern industrial research was focused on cryogenic treatment of ferrous alloys, especially on cutting tools as they exhibit longer wear and higher durability [1]. This treatment is extended to even for nonferrous alloys to improve their hardness properties [2]. It is well known that the cryogenic treatment enhances metallurgical properties of most of the metals, which in turn improve various strengths of the treated parts [3]. It creates denser molecular structure of the metals and alloys resulting in a larger contact surface area that reduces friction, heat and wear. This work was also extended on metal matrix composites (MMCs) to enhance the tensile properties [4]. Cryogenic treated MMCs showed exceptionally high hardness values, which are attributed to the formation of ternary phases at the temperatures of consolidation [5]. However, a few researchers focused on the effect of cryogenic treatment on mechanical and wear properties of MMCs but none of the work is focused on optimization of cryogenic process parameters on the basis of the best properties of MMCs.

It is known that the full economic and technical potential of any manufacturing process can be harnessed only when the process is carried out with the optimum parameters. One of the most important robust optimization techniques is Taguchi method [6]. The Taguchi approach enables a comprehensive understanding of the individual and combined process parameters from a minimum number of simulation trials. The cryogenic parameters are important to study the influence on wear properties of Al/$Al_2O_3$ composites. Hence this investigation was focused on cryogenic parameters of MMCs at three levels and three factors each. In this paper, Taguchi method was used for optimization of cryogenic parameters such as cryogenic temperature, duration of cryo-treatment and wt. % of $Al_2O_3$ factors.

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## II. EXPERIMENTALPROCEDURE

The Al 6061 alloy (matrix material) and $Al_2O_3$ 30-50μm 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 particle was chosen as commercial $Al_2O_3$ with 99.5% purity.

**Table 1 Chemical Composition of Al 6061**

| | |
|---|---|
| Mg | 0.92 |
| Si | 0.76 |
| Fe | 0.28 |
| Cu | 0.22 |
| Ti | 0.1 |
| Cr | 0.07 |
| Zn | 0.06 |
| Mn | 0.04 |
| Be | 0.003 |
| V | 0.01 |
| Al | Bal |

The composites were prepared by adding 0, 10 & 20 wt.%. of $Al_2O_3$ by liquid metallurgy technique. 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 gas for about 3 to 4 min. The resulting mixture was tilt poured into preheated permanent moulds. 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 glass wool insulator after placing the samples in the chamber. This chamber was progressively immersed in a liquid nitrogen reservoir. The samples temperature was monitored by a K type thermocouple which was used to operate a step motor to lower the samples in order to maintain a temperature decline at the rate of 1 °C/min. it took about 4 h to reach the deep cryogenic temperature of -196°C. Specimens were held at cryogenic temperature for 0, 25 and 50 h and then slowly brought up to + 25 °C. The cryogenic procedure is followed as per Kaveh Meshinchi et.al. [7]. 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 Olympus metallurgical microscope, fitted with a camera. The specimens were washed with distilled water followed by acetone and dried thoroughly.The wear specimens (6 mm dia and 15 mm length) were tested under dry (unlubricated) conditions in accordance with ASTM G99 standards using a pin-on-disc sliding wear testing machine. The apparatus consists of an EN24 steel (BHN 229) disc of diameter 200 mm used as counter-face. The test sample was clamped in a holder and held against the rotating steel disc. The test was conducted at constant speed and load to find the influence of cryogenic treatment on wear rate.

## III. EXPERIMENTALDESIGN

In order to observe the influencing degree of cryogenic process parameters in the wear rate of $Al_2O_3$ /Al MMCs, three parameters namely; (1) cryogenic temperature; (2) Treatment duration and (3) particle wt.%, each at three levels were considered and are listed in Table 2.

**Table 2. Cryogenic treatment control parameters**

| Factors | Control factor | DOF | Level 1 | Level 2 | Level 3 |
|---|---|---|---|---|---|
| A | Cryogenic temperature, °C | 02 | -100 | -150 | -200 |
| B | Duration of treatment, h | 02 | 0 | 25 | 50 |
| C | Wt. % of $Al_2O_3$ | 02 | 0 | 10 | 20 |

Maintain the cryo-treatment parameters as constants to enable the study of the effect of cryogenic temperature; (2) Treatment duration and (3) particle wt.%, in the wear result. The degrees of freedom for three parameters in each of three levels were given in Table 2. Table 3 indicates the parameters used and the results of wear test. A three levels L9, $3^4$ orthogonal array with nine experimental runs was selected. The total degree of freedom is computed. In this research nine experiments were conducted at different cryogenic parameters, and then the specimens were machined and tested for wear behaviour.

**Table 3. Experimental observation**

| Exp . No | Cryogenic temp. °C | Treatment duration, h | Wt.% of $Al_2O_3$ | Average Wear rate $mm^3/km$ | Standard Deviation | S/N ratio |
|---|---|---|---|---|---|---|
| | A | B | C | | | |
| 01 | -100 | 0 | 0 | 0.8071 | 0.1101 | 8.65 |
| 02 | -100 | 25 | 10 | 0.1493 | 0.025 | 7.76 |
| 03 | -100 | 50 | 20 | 0.0458 | 0.0351 | 11.51 |
| 04 | -150 | 0 | 20 | 0.1841 | 0.0323 | 7.557 |
| 05 | -150 | 25 | 0 | 0.7046 | 0.0471 | 11.752 |
| 06 | -150 | 50 | 10 | 0.1647 | 0.0209 | 8.959 |
| 07 | -196 | 0 | 10 | 0.2963 | 0.0336 | 9.458 |
| 08 | -196 | 25 | 20 | 0.0409 | 0.0309 | 12.21 |
| 09 | -196 | 50 | 0 | 0.5632 | 0.0776 | 8.606 |

The S/N ratios were computed for wear rate in each of the nine trial conditions and their values are given in Table 3.

## IV. RESULTS AND DISCUSSION

### 4.1 Micro-structural studies

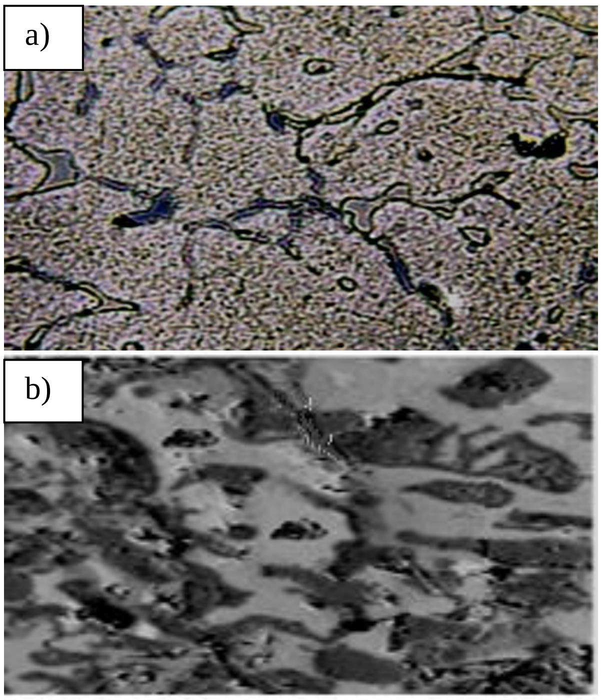



**Fig. 1. Microstructure of the surface of a) as cast specimen (A1, B1 & C1) and b) cryogenically treated Al/$Al_2O_3$ MMCs (A2, B3 & C3) respectively.**

Microstructure of as cast (A1, B1 & C1) and cryogenic treated Al/20%$Al_2O_3$ MMCs specimen (A2, B3 & C3) are given in the Fig. 1(a) and 1(b) respectively. 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 wear properties. 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.

### 4.2 Optimization based on S/N ratio

Optimization of the process parameters was performed using the results from wear test. The column in the design matrix (to which factors were assigned) and the experimental levels for the factors, are the same as in the case design for a wear test. Taguchi method stresses the importance of studying

S N Ratio

the response variation using the signal-to-noise (S/N) ratio, resulting in minimization of quality characteristic variation due to uncontrollable parameter. The wear rate were considered the quality characteristic with the concept of "the larger the better". The S/N ratio used for this type response is given by [8]:

$$\frac{S}{N} = 10\ log_{10}\left(\frac{\bar{x}^2}{\sigma^2}\right) \text{ in decibels}$$

For S/N ratios, Fig. 2 shows that the factor B (duration of treatment) have strong effects, factor C (% of reinforcement) is the next and Factor A appears to be the last. From the above calculations it is apparent that the maximum S/N ratio is desirable in order to have the highest wear rate, hence the following assignments should be made

Factor A (cryogenic temperature) at level 2 (-150 °C),
Factor B (treatment duration) at level 3 (50 h)
Factor C (% of reinforcement) at level 3 (20 wt.% of $Al_2O_3$)

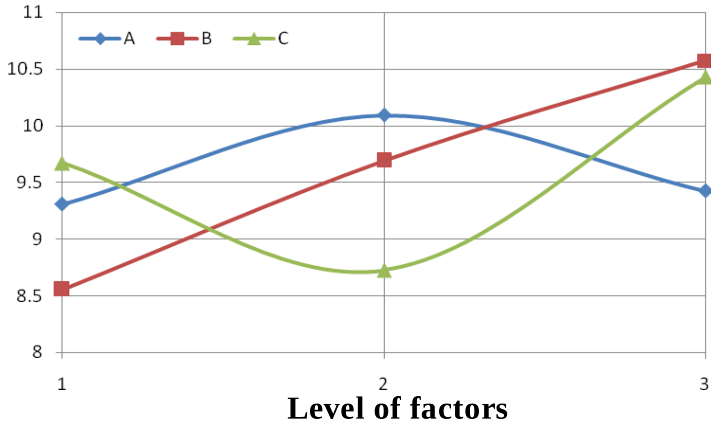



**Fig. 2 Plot of factor effects on S/N ratio**

### *4.3* Optimization on the basis of ANOVA

The cryogenic treatment parameters, namely cryogenic temperature (A), duration of treatment (B), and particles wt.% (C) were assigned to the 1st , 2nd and 3rd column of L9 $3^4$array, respectively. The $4^{th}$ column was assigned as error (E), and was considered randomly. The S/N ratios were computed for wear rate in each of the eight trials conditions and their values are given in Table 3. Computation scheme of ANOVA for three level factors as per standard is given in table4. In order to study the contribution ratio of the process parameters, ANOVA was performed for wear rate as given in Table 4.

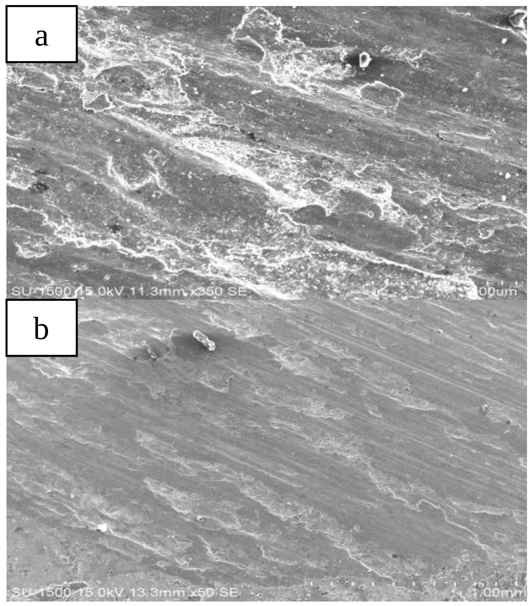
**Table 4- ANOVA Table**

| Factor | | A | B | C | E | Total |
|---|---|---|---|---|---|---|
| Sum at factor level | 1 | 1.288 | 1.288 | 2.075 | 1.013 | 2.205 |
| | 2 | 0.314 | 0.895 | 0.610 | 0.897 | |
| | 3 | 0.604 | 0.774 | 0.271 | 1.047 | |
| Sum of square difference | | 1.499 | 0.433 | 5.515 | 0.037 | 7.484 |
| Degree of freedom | | 02 | 02 | 02 | 02 | 08 |
| % Contribution | | 20.03 | 5.78 | 73.69 | 0.50 | 100 |
| Optimum Level | | 02 | 03 | 03 | | |
| | | A2=150 °C | B3 = 50 h | C3 = 20% | | |

From Table 4, it can be seen that the second level of factor (A) give the lowest summation (i.e A2, which is sum of responses at temperature -150 °C). The lowest summation for factor (B) is at the third level (i.e B3, which is 50 hours) and the lowest summation for factor (C) is at the third level (i.e C3, which is 20% wt. %). These predicted parameters are not used in the cryogenic treatment which is indicated in Table 2 and Table 3. Hence conducted experiments at the predicted parameters, ie, cryogenic temperature of -150 °C (A2), duration of treatment 50 hrs (B3) and $Al_2O_3$ wt. % of 20 % (C3) and tested the resulted specimen by wear test. The resulted wear rate was 0.153 $mm^3/m$, which is lower than the wear rate values given in Table 3. These results have proved the success of both S/N ratio method and Taguchi method in the prediction of the optimum parameters for lower wear rate. The last but one row in Table 4 shows the percentage contribution of each factor on the total variation indicating their degree of influence on the result. One can observe from the ANOVA table that the % contribution of cryogenic temperature (= 20.03%), treatment duration (= 5.783%) and the reinforcement (= 73.69%) has great influence on the wear resistance. The reinforcement is influencing comparatively high which indicates that there is greater appreciable increase in wear resistance by increasing the reinforcement content from 0 to 20 wt. %. The various researchers (9, 10) have the opinion that as the reinforcement content increases, the dry sliding wear resistance increases. The quantity of increase in wear resistance will be less with further increase in reinforcement content. Sannino and Rack (11) recognized that an increase in hard ceramic reinforcement in discontinuously reinforced aluminum composites up to approximately 20 volume fraction improves the abrasion and fretting wear resistance.

### 4.4 Worn out surface studies

a




b

***Fig.3 Typical wear surface of Al/ 20% $Al_2O_3$ MMCs at applied load of 20 N and sliding speed of 1.5 m/s (a) A1, B1 & C1 (higher wear rate), (b) A2, B3 & C3 (at lower wear rate)***

The Fig. 3(a) and (b) shows the SEM of worn surfaces of the ascast specimen (A1, B1 & C1) and cryogenically treated composites (A2, B3 & C3) respectively. The large non distinct parallel and continuous grooves are formed in ascast specimen and a very large quantity of plastic deformation can also be observed from the Fig.3 (a). With increase in % reinforcement and deeper cryogenic temperature the large distinct grooves will reduce to fine scratches as shown in Fig. 3(b). The damaged spots have been observed in the form of crater. The worn out surfaces have proved the success of both Taguchi and S/N ratio method.

## V. CONCLUSIONS

Taguchi's and S/N ratio methods used to determine the optimal process parameters which minimize the number of experimentations to be conducted to determine the wear properties of cryogenically treated $Al/Al_2O_3$ composites were found fruitful. For this purpose, concepts like orthogonal array, S/N ratio and ANOVA were employed. After determining the optimum process parameters, one confirmation experiment was conducted. The optimum level of cryogenic process parameters to obtain good wear resistance of Al /Al2O3 are 20 % wt. of $Al_2O_3$ particles, cryogenic temperature of -150 °C and treatment duration of 50 hours. The optical microscope, experimental results and worn out surface justify both Taguchi and S/N ratio optimization techniques. Wt. % of $Al_2O_3$ is contributed on wear resistance (73%), cryogenic temperature (20.3%) and cryogenic treatment duration (5.783 %). The pooled error associated with the ANOVA is 0.5 % for the factors.

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