INFLUENCE ON MECHANICAL PROPERTIES BY CRYOGENIC TREATMENT ON ALUMINIUM ALLOY 7075

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***Abstract* — Al-Mg alloys are extensively used in transport applications, including marine, automotive and aviation, due to their high strength-to-density ratio. Cryorolling is one of the important deformation processes to produce sheets with high strength. Because of their strength and light weight, Al-Mg alloys is also desirable in other fields. Rock climbing equipment, bicycle components, inline skating-frames and hang glider airframes are commonly made from 7075 aluminum alloy. They are also used for the manufacture of M16 rifles for the American military. In the present work, formability of cryorolled AA7075 alloy sheets was characterized. Sheet samples were cold rolled and cryorolled with 30% reduction in thickness and mechanical properties were compared. Cryogenic treatment was done on the sheet samples of AA7075 in order to improve their mechanical properties without loss in their strength with respect to reduction in thickness.**

I.INTRODUCTION

Aluminum alloys with a wide range mechanical properties are used in various engineering applications. Due to their lower weight and excellent corrosion resistance, Al alloys are extensively used in aerospace industry. In the aerospace industry, there is a continuing trend towards use of aluminum alloys in their construction. However, one of the limitations of aluminum alloys is lower strength and formability

It is strong, with a strength comparable to many steels, and has good fatigue strength and average machinability, but has less resistance to corrosion than many other Al alloys.

ITEM	PERCENTAGE
ALUMINIUM	BALANCE
CHROMIUM	0.18-0.28
COPPER	1.2 – 2
MAGNESIUM	2.1 - 2.9
ZINC	5.1 - 6.1
MANGNESE	0.3 max
SILICON	0.4 max
TITANIUM	0.2 max

Table: Composition of Aluminium Alloy

A. Rolling:

Rolling is a metal forming process in which metal stock is passed through one or more pairs of rolls to reduce the thickness and to make the thickness uniform. The concept is similar to the rolling of dough. Rolling is classified according to the temperature of the metal rolled.

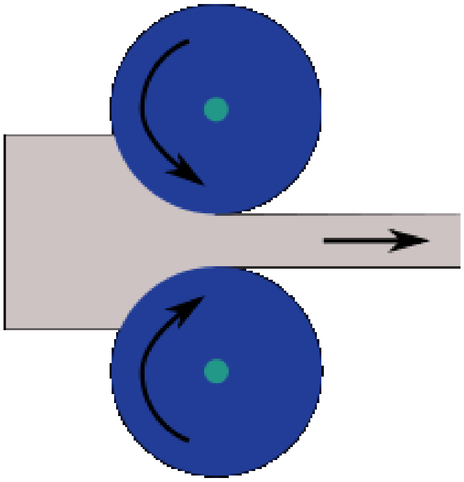


Fig. Rolling Process

B. *Types of rolling:*

Hot rolling - If the temperature of the metal is above its recrystallization temperature, then the process is known as hot rolling.

Cold rolling - If the temperature of the metal is below its recrystallization temperature, the process is known as cold rolling.

C. *Cryogenic treatment:*

"Cryogenics" stems from Greek and means "the production of freezing cold"; however the term is used today as a synonym for the low-temperature state. It is not well-defined at what point on the temperature scale refrigeration ends and cryogenics begins. Cryogenics typically involves a deep-freezing process, usually one that takes object down below 240 degrees Fahrenheit and changes the molecular alignment of the material structure. This change creates the new property. Cryogenic process has been researched and developed by universities and NASA since the mid-sixties after NASA discovered that deep space exploration vehicles had improved their structural integrity due to extended exposure to cryogenic temperature. Cryorolling, is one of the potential techniques to produce nanostructured bulk materials from its bulk counterpart at cryogenic temperatures. It can be defined as rolling that is carried out at cryogenic temperatures. Nanostructured materials are produced chiefly by severe plastic deformation processes. The majority of these methods require large plastic Deformations. In case of cryorolling, the deformation in the strain hardened metals is preserved as a result of the suppression of the dynamic recovery. Hence large strains can be maintained and after subsequent annealing, ultra-fine-grained structure can be produced.

D. *Types of cryogenic treatment*

- Shallow Cryogenics, made the objects to temperature of approximately-120 °F.
- Flooding, takes the component to -120 °F, then the chamber is flooded with liquid nitrogen.
- Deep Cryogenics Treatment, Subjects the objects to the temperature of approximately -300 °F.

II. PROPOSED METHODOLOGY

A. *Cryogenic Treatment*

Cryogenic Treatment is a material science and involves the process of reducing the temperature of components over an extended period of time to extreme cold levels, usually slightly below -250°C. A cryogenic treatment is the process of treating work pieces to cryogenic temperatures (i.e. below −190 °C (−310 °F) to remove residual stresses and improve wear resistance on steels. In addition to seeking enhanced stress relief and stabilization, or wear resistance, cryogenic treatment is also sought for its ability to improve corrosion resistance by precipitating micro-fine eta carbides, which can be measured before and after in a part using a quantimet.

On the other hand, cryogenic treatment, also known as subzero treatment, is a very old process that has been used widely for high precision parts and objects and especially for the ferrous materials mentioned earlier (Sendooran and Raja 2011). Subjecting materials to extreme cold hardens and strengthens; this method has been used for centuries (Bensely et al. 2007). Now cryogenic treatment is widely used in the automotive, aerospace, electronic and mechanical engineering industries to improve mechanical strength and the dimensional stability of various components (Zhirafar et al. 2007). For the past few years, in order to improve properties, a cryogenic treatment for nonfer- rous metals such as aluminium and magnesium alloys has been used

(Kaveh et al. 2009). The mechanical properties and microstructure of metals and alloys in cryogenic treatment have drawn the attention of researchers. Lulay et al. (2002) and Jiang et al. (2009) showed the beneficial effects of cryogenic treatment on nonferrous metal aluminium. When considering the wear performance of copper alloy, cryogenic treatment yields the least significant changes (Guozhi et al. 2010). However, Woodcraft and Adam (2005) showed a significant improvement in the mechanical properties of the strength, hardness, and toughness of aluminium alloy when subjected to cryogenic treatment. This has led to the idea of analyzing individual alloys' properties when MMCs undergo cryogenic treatment. The present work

Structure of Materials being treated; dependent on the composition of the material it performs three things:

- Turns retained austenite to martensite
- Refines the carbide structure
- Stress relieves

Cryogenic treatment of ferrous metals converts retained austenite to martensite and promotes the precipitation of very fine carbides. Most heat treatments at best will leave somewhere between ten and twenty percent retained austenite in ferrous metals, because austenite and martensite have different crystal structures, there will be stresses built into the crystal structure where the two co-exist. Cryogenic processing eliminates these stresses by converting the majority of the retained austenite to martensite. An important factor to keep in mind is that Cryogenic processing is not a substitute for heat treating if the product is poorly treated cannot help in changing the austenite to martensite. If the product is over heated during remanufacture or over stressed during use, the temper of the steel which is developed during the heat treatment process may be destroyed, rendering the Cryogenic process useless by default. Cryogenic processing will not in itself harden metal like quenching and tempering. It is an additional treatment to heat treatment. This transformation itself can cause a problem in poorly heat treated items that have too much retained austenite. It may result in dimensional change and possible stress points in the product being treated. This is why Cryogenic industries will not treat, poorly heat treated items. The Cryogenic metal treatment process also promotes the precipitation of small carbide particles in tool steels and suitable alloying metals.

The fine carbides act as hard areas with a low co-efficient of friction in the metal that greatly adds to the wear resistance of the metals. Japanese studying the role of carbides in the wear resistance improvements of tool steel by Cryogenic treatment, concluded the precipitation of fine carbides has more influence on the wear resistance increase than does the removal of the retained austenite. The process also relieves residual stresses in metals and some forms of plastics. This has been proven by field studies conducted on product on high impact scenario where stress fractures are evident. Cryogenic treatment refers to subjecting materials to very low temperatures. This process is not limited in the application to metals, but can also be applied to a wide range of materials with different results. Many commercial industries have extolled the benefits of Cryogenic treatment, but few have extensively studied the mechanism of Cryogenic treatment.

Several different cryogenic processes have been tested by the researchers. These involve a combination of deep freezing and tempering cycles. Generally they can be described as controlled lowering of temperature from room temperature to the boiling point of liquid nitrogen (-196^{o}C), maintenance of the temperature for about twenty four hours, followed by a controlled raising of the temperature back to room temperature. Subsequent tempering process may follow. In tool steels, this treatment affects the material in two ways. Firstly, it eliminates retained austenite and hence increases the hardness of the material. Secondly this treatment initiates nucleation sites for precipitation of large number of very fine carbide particles, resulting in an increase in wear resistance.

B. Advantages:

- In Cryorolling, the strain hardening is retained up to the extent to which rolling is carried out. This implies that there will be no dislocation annihilation and dynamic recovery. Whereas in rolling at room temperature, dynamic recovery is inevitable and softening takes place.

- The flow stress of the material differs for the sample which is subjected to cryorolling. A cryorolled sample has a higher flow stress compared to a sample subjected to rolling at room temperature.
- Cross slip and climb of dislocations are effectively suppressed during cryorolling leading to high dislocation density which is not the case for room temperature rolling.

- The corrosion resistance of the cryorolled sample comparatively decreases due to the high residual stress involved.
- The number of electron scattering center increases for the cryorolled sample and hence the electrical conductivity decreases significantly.
- The cryorolled sample shows a high dissolution rate.

III. EXPERIMENTAL PROCEDURE

A. *Procedure 1*

First the properties of aluminium alloy were studied in this study, wrought aluminum alloy AA7075 has been used for producing thin sheets by cryorolling. It is Mg alloy with very small amounts of Mn and Cr. Additionally, this particular alloy does not show ductile to brittle transformation at very low temperatures, to brittle transformation at very low temperatures, which makes it possible to enhance its strength by rolling at subzero temperatures.

Fig : Base Metal

AA 7075-T651:

T651 temper 7075 has an ultimate tensile strength of at least 67,000–78,000 psi (462–538 MPa) and yield strength of 54,000–67,000 psi (372–462 MPa). It has a failure elongation of 3–9%. The 51 suffix has no bearing on the heat treatment but denotes that the material is stress relieved by controlled stretching.

B. *Tensile Test*

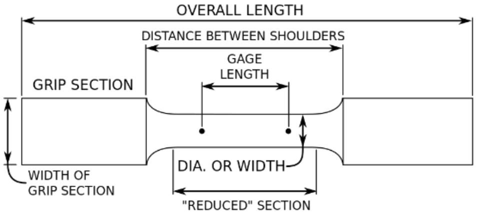
A tensile test, also known as tension test, is probably the most fundamental type of mechanical test you can perform on material. Tensile tests are simple, relatively inexpensive, and fully standardized.

Tensile specimens made from an aluminum alloy. The left two specimens have a round cross-section and threaded shoulders. The right two are flat specimen designed to be used with serrated grips. A tensile specimen is a standardized sample cross-section. It has two shoulders and a gauge in between. The shoulders are large so they can be readily gripped, whereas the gauge section has a smaller cross-section so that the deformation and failure can occur in this area.

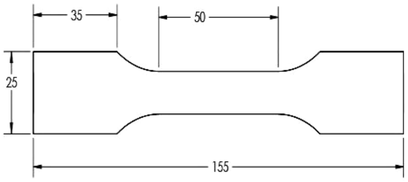
The shoulders of the test specimen can be manufactured in various ways to mate to various grips in the testing machine (see the image below). Each system has advantages and disadvantages; for example, shoulders designed for serrated grips are easy and cheap to manufacture, but the alignment of the specimen is dependent on the skill of the technician. On the other hand, a pinned grip assures good alignment. Threaded shoulders and grips also assure good alignment, but the technician must know to thread each shoulder into the grip at least one diameter's length, otherwise the threads can strip before the specimen fractures.

The following standards is used for cutting the required specimen

- ASTM E8/E8M-13: "Standard Test Methods for Tension Testing of Metallic Materials" (2013).
- ISO 6892-1: "Metallic materials. Tensile testing. Method of test at ambient temperature" (2009).
- ISO 6892-2: "Metallic materials. Tensile testing. Method of test at elevated temperature" (2011).
- JIS Z2241 Method of tensile test for metallic materials.

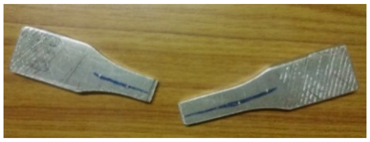


- Thus the specimen was machined to a dimensions of 155x25x6mm as required by the ASTM standards.



Base Metal Tensile Testing Dimensions

Base Metal Machined



Base Metal Tested

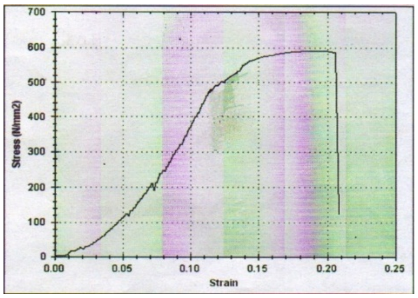
Description	
Specimen Shape	Flat
Specimen Type	Aluminium
Specimen Description	AA7075 T6
Specimen Width	12.6 mm
Specimen Thickness	6.5 mm
Initial Gauge Length	50 mm
Final Specimen Width	0 mm
Final Specimen Thickness	0 mm
Final Gauge Length	53.2 mm
Specimen C S Area	81.9 mm^2

Base Metal Data

Load at yield	39.9 kN
Yield Stress	487.179 N/mm^2
Load at Peak	48.340 kN
Tensile Strength	590.232 N/mm^2
Elongation	6.40%

Tensile Test Data

- The following is the stress strain graph obtained for the above base metal when tested.



Stress – Strain Graph

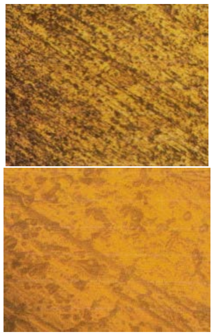
C. *Hardness Test*

Hardness is a characteristic of a material, not a fundamental physical property. It is defined as the resistance to indentation, and it is determined by measuring the permanent depth of the indentation. More simply put, when using a fixed force (load) and a given indenter, the smaller the indentation, the harder the material. Indentation hardness value is obtained by measuring the depth or the area of the indentation using one of over 12 different test methods.

The Rockwell hardness test method, as defined in ASTM E-18, is the most commonly used hardness test method. The Rockwell test method is used on all metals, except in condition where the test metal structure or surface conditions would introduce too much variations; where the indentations would be too large for the application; or where the sample size or sample shape prohibits its use. The most common indenter type is a diamond cone ground at 120 degrees for testing hardened steels and carbides. Softer materials are typically tested using tungsten carbide balls ranging in diameters from 1/16 in up to 1/2 in. The combination of indenter and test force make up the Rockwell scale. These combinations make up 30 different scales and are expressed as the actual hardness number followed by the letters HR and then the respective scale. A recorded hardness number of HRC 63 signifies a hardness of 63 on the Rockwell C scale. Higher values indicate harder materials such as hardened steel or tungsten carbide. These can have HRC values in excess of 70 HRC.

D. Microstructure Test

Microstructure is defined as the structure of a prepared surface or thin foil of material as revealed by a microscope above 25× magnification. The microstructure of a material (which can be broadly classified into metallic, polymeric, ceramic and composite) can strongly influence physical properties such as strength, toughness, ductility, hardness, corrosion resistance, high/low temperature behavior, wear resistance, and so on, which in turn govern the application of these materials in industrial practice. Microstructure at scales smaller than can be viewed with optical microscopes is often called ultrastructure or nanostructure.



Microstructure of Base Metal before Rolling

IV. EXPERIMENTAL PROCEDURE 2

Since high reductions in thickness are essential for achieving ultra-fine grain structure, 6mm thick sheet sample of size 150mmX100mm were rolled at room temperature conditions by using a 2 high rolling mill with 30% reduction so that the final sheets were produced from 6mm to 4.2mm thickness and were then treated with liquid nitrogen at cryogenic temperatures.

Aluminum alloys strengthened by cold or cryorolling exhibit reduction of ductility which would make the material unsuitable for forming applications. Therefore, a suitable heat treatment was given to the cold rolled sheets to improve ductility without sacrificing too much on strength. A cryogenic treatment with temperature below -190°C for 60 min was done with the help of liquid nitrogen bath to achieve the desired combination of strength and ductility. Cryorolled metal shows the increase in strength as it is mentioned in the desirable properties of 7075 alloy and it is also produce greatest spring back during forming process. The suppression of dynamic recovery and accumulation of higher dislocation density contribute to improved mechanical properties of this aluminum alloy. Accordingly, the cryogenic deformation would require less plastic deformation for achieving UFG structure, compared to the severe plastic deformation processes at ambient or elevated temperatures.

The cryorolled Al alloy investigated under HCF regime of intermediate to low plastic strain amplitudes has shown the significant enhancement in fatigue strength as compared to the coarse grained bulk alloy due to effective grain refinement. The reasons behind such crack growth retardation is due to diffused crack branching mechanism, interaction between a propagating crack and the increased amount of grain boundaries, and steps developed on the crack plane during crack-precipitate interaction at the GB due to ultrafine grain formation.

Fig. Rolled Base Metal

These plates were rolled using two high rollers with a determined load in order to reduce its thickness by 30%. This rolling process was done at KMC Aluminium works, Coimbatore. The impact of rolling of the base metal dimensioned 150mmx100mmx60mm increased it dimensions by 20% and the final when measured was 230mmx100mmx4.2mm.

A. *Tensile Test*

Tensile samples of CLR sheets were tested on Universal Testing Machine at room temperature. The samples were prepared by milling according to ASTM E8 standards. The load elongation data was obtained from the tensile tests on cryorolled and cold rolled specimens. Based on the load-elongation data, engineering stress engineering strain were plotted were shown

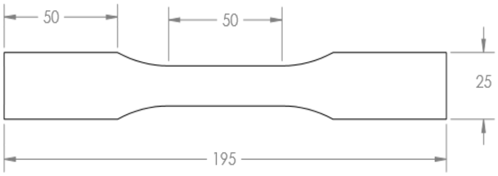


Fig. ASTM standard for tensile

Load at yield	26 kN
Yield Stress	476.19 N/mm^2
Load at Peak	33.820 kN
Tensile Strength	619.414 N/mm^2
Elongation	1.20%

Table : Tensile Test Output Results

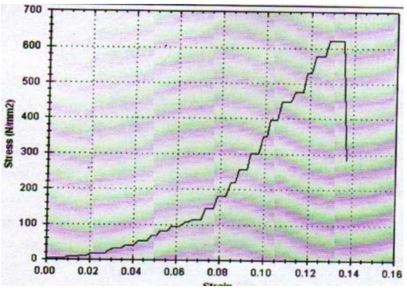
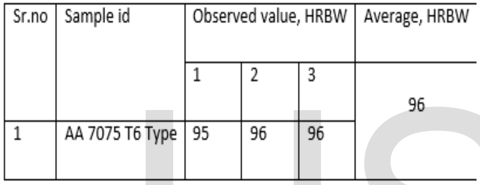


Fig. Stress Strain Graph Post Rolling

B. *Hardness Test*

The hardness tests of the material is an important property that affect the wear strength of the materials. It was observed that the improvement in the properties of aluminium alloy after cryogenic treatment was negligible when compared to the other materials like the tool steels where significant improvement in properties are noticed. The increase in Hardness is approximately within 5% for all the Aluminium alloys after the cryogenic treatment.



Sr.no	Sample id	Observed value, HRBW			Average, HRBW
		1	2	3	96
1	AA 7075 T6 Type	95	96	96	

The hardness values in as rolled condition are also shown for comparison. The hardness values of as cryorolled material after 30% reduction are 6% higher than that of the cold rolled sheets. The enhancement of hardness of cryorolled sheets is due to higher dislocation density [Panigrahi & Jayaganthan, 2008] pure metals and alloys at cryogenic temperatures suppresses dynamic recovery and the density of accumulated dislocations reaches higher levels with the increasing number of cryorolling passes [Wang et al., 2003]. The hardness decreased with annealing at 250 C.

C. *Microstructure Test*

The microstructures of the initial base metal as received reveals coarse equiaxed grains. The microstructures in cryorolled and cold rolled condition with 30% reduction. The substructure formation with an unclear cell network has been found. But the microstructure of cryorolled sample after 30% reduction shows ultra-fine grain structure possibly with grain size less than1 micron. The grain refinement and grain size less than1 micron. The grain refinement and increased dislocation density contribute to improved mechanical properties such as higher strength and mechanical properties such as higher strength a hardness as compared to that of their bulk materials. The microstructure of cryorolled sample annealed at 250° C after 30% reduction reveals bigger grains than in normal base metal and much finer than in the initial as received microstructure.

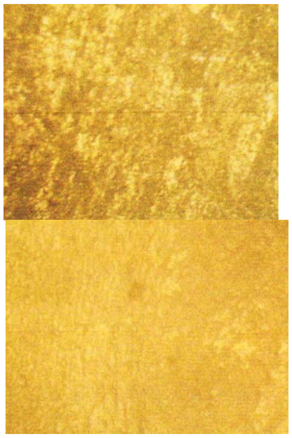


Fig .Microstructure Image after Rolling

The above results shows that the structure are more refined when compared to the base metal before being rolled. The image was taken was taken at a magnifying level 100X and 400X using Kellers reagent.

V. RESULTS AND DISCUSSION

This chapter presents the results of different Mechanical properties of Aluminium alloy, cold rolled to a certain thickness and cryogenically treated Aluminium alloy after annealing for 1 hour and then compare the performance of mechanical properties of the base metal aluminium alloy and cryogenically treated alloy. Effect of cold rolling and cryorolling on the Mechanical properties of Aluminium alloy.

A. *Tensile Test*

The maximum value of UTS was obtained in specimens subjected to cryorolling for 1 hours, which is 619.414 N/mm^2. This shows an increase of 30% over the base metal alloy value 590.232 N/mm^2. This can be attributed to the precipitate particles of the alloying elements distributed in the matrix.

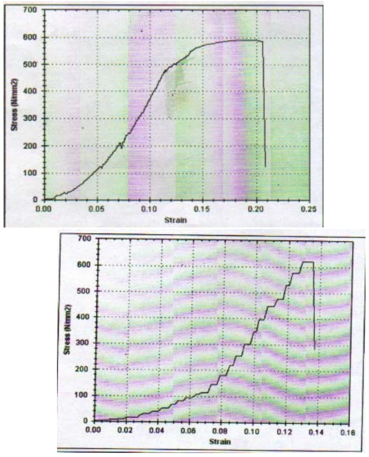


Fig. Comparison of Tensile Tests

The percentage of elongation has decreased when the specimen is subjected to 1 hour of cryogenic treatment. The maximum decrease of percentage of elongation obtained is 75% over base metal value of 6.40% to 1.20% elongation.

B. *Hardness Test*

The below results is Rockwell Hardness being done on the base metal of aluminium alloy 7075. The initial average results tabulated was 84 BHN. These results were generated after cold rolling the base metal at a specific temperature. The base metal after being rolled with 30% reduction and then being treated with cryogenic treatment and cryogenic temperature. The dimensions of the plate has been changed due to the stress applied by the two high rollers and the load applied. The base metal after being rolled was 230mmX100mmX42mm from its original dimensions. But when it was cryogenically treated its hardness had been increased by 13% from its original value. The average hardness after being tested was 96 BHN.

TEST RESULTS		
Observed value HRBW	BEFORE ROLLING	AFTER ROLLING
	Sample AA 7075	Sample AA 7075
1	85	95
2	83	96
3	85	96
AVERAGE	87	96

Table: Comparison Table of Hardness Results

C. *Microstructure Test*

The details of the microstructural examination are carried out on Aluminium-Magnesium-Silicon alloy. As cryogenically treated specimens are presented in the following figures:

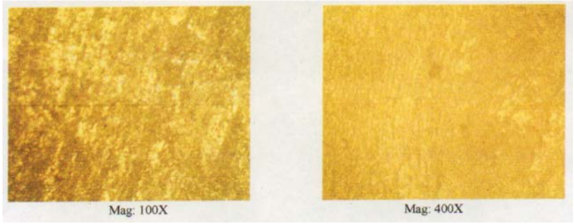


Fig. Microstructural View after Rolling

Specimen for micro-structure study was prepared using standard procedure for specimen preparation, they were etched using Keller's agent. Microstructure examination was carried out using an optical microscope. The micro-structure of the base metal sample is shown in which shows varying size of precipitates randomly distributed in the matrix.

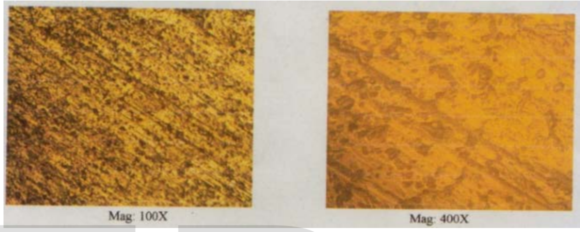


Fig Microstructural view before Rolling

It shows the optical micrographs of both etched and un-etched samples of cold rolled and cryorolled specimens for 1 hour. Precipitants are predominantly seen dispersed in the matrix. The precipitant particles appear to be in globular form for specimens solutionized for 1 hours. In the case of samples of cryorolled samples for 1 hour the precipitates appear to be a matrix of globules as well as platelets.

VI. CONCLUSION

From the results of the investigation carried out on the rolling treatment and cryogenic treatment of Al- Mg- Si alloy, the following conclusions are made:

- The considerable improvement of 13.42% in Ultimate tensile stress was achieved by subjecting Al-Si-Mg alloy specimens to solutionizing and age hardening. A further improvement in Ultimate tensile stress was observed at all aging times when subjecting the sample to deep cryogenic treatment.
- Percentage elongation of both cold rolled and cryogenic treated specimens decreases 20% with increase in aging time.
- Comparing the hardness of specimens, cold-rolled and cryorolled specimens exhibited

much higher hardness of 13%. Hardness increases with increase in aging time. Further much superior hardness is noticed in heat treated specimens subjected to cryogenic treatment.

- A significant increase in wear resistance is observed in cryorolled specimens than the base metal. Further improvement in wear
- Aluminum-Silicon-Magnesium specimens to cryogenic treatment.
- Cryogenic treatment which is quite new addition to the industry is found to further

resistance is achieved upon subjecting heat treated specimens to deep cryogenic treatment.

- From this work one can conclude that a considerable improvement in mechanical properties of Aluminium alloy like Proof stress, Ultimate tensile stress, Hardness and wear resistance are achieved by subjecting
- Enhance the Mechanical properties considerably. Hence Cryogenic treatment is recommended as an effective means of roving Mechanical properties of Al-Si-Mg alloys.

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