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Effect of cryogenic treatment on mechanical properties and microstructure of aluminium 6082 alloy

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A B S T R A C T

Solution heat treatment and Cryogenic treatment of Al6082 aluminium alloy were experimentally carried out at Temperature 520 °C and −183 °C temperature by using muffle furnace and by dipping the specimen in liquid nitrogen solution bath respectively. The soaking time is varied from 10 min to 24 h in four step (10 min, 30 min, 60 min and 24 h). The variation in the tensile strength and hardness were evaluated. The tensile strength and hardness increases as a function of cryogenic duration. The results were compared with as received and solution treated specimen. The microstructure of the solution and cryo-treated specimen were studied by optical microscope. The grain size refinement was recorded in cryo-treated specimen. The grain size refinement increases with increase in the duration of cryo-treatment. The fractography of broken tensile specimen were done using SEM to discuss about ductile and brittle fracture.
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1. Introduction

The production of any component possesses a series of operations. Each operation alters the microstructure of end product and in result mechanical properties changes [1–7]. In some application, high strength to weight ratio is an important and demanding factor for the selection of any material. In this category, aluminium alloys become very popular as compared to other materials including ferrous alloys. Various aluminium alloys are available and classified as series of Al. Each series of Al composed of unique composition of elements, which provided various unique properties for industrial and large commercial use. For aerospace industries, Al alloys are prime candidate due to their ease manufacture, modest specific strength and low cost. It also provides increased fuel efficiencies and payloads for aerospace industries. It's always required to enhance the performance of existing alloys by development of more advanced materials with high specific properties. Later on composites and titanium alloys are used in in aerospace industry, but could not get that much of popularity as of Al alloys due to unease in manufacturing and high cost.

Aluminium alloys are mainly used in the manufacturing of heat exchanging apparatus of air conditioners [8]. There is a need to increase the properties of aluminum alloys at elevated temperature for high temperature applications. Al 5XYZ series are widely used in the manufacture of pressure vessels, aerospace industries etc. requires increased performance [9]. In this process there is a need to increase the properties of conventional materials like Aluminium alloys without sacrifice of any other property. This has opened the door for the low temperature treatment of materials called as cryogenic. In cryogenic treatment, component gradually cools to predefined temperature, holding it at that temperature for predefined time and then progressively leading it back to the room temperature. The aim of cryogenic treatment is to obtain an improvement hardness and wear resistance. With help of cryogenic treatment, component's life, thermal properties and machining can be enhanced [10].

Senthilkumar et al. [11] studied the effect of cryogenic treatment on mechanical properties and residual stresses of Al alloy. They slowly cooled material without thermal shocks nearly around −184 °C, hold the material for 24 h at this temperature for soaking for a period of 24 h and reheated bit by bit. In results they obtained that the residual stresses was reduced by up to 9ksi in the base metal. Gentle increase in fatigue, micro hardness and tensile properties were also observed. The effect of cryogenic treatment on the

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room temperature strength, hardness, and toughness of aluminium 7075-T651 was investigated by Cui et al. [12]. The treatment was performed in two variant in first, test specimens was kept in a commercial cryogenic freezer (−196 °C) 2 h; and in second it was kept for 48 h. For identifying and evaluate any time independent effect and soaking effect in the cryo. Treatment, 2 h and 48-h treatment was conducted. It has been observed that the effect of 48-h cryogenic treatment was not too significant on the basic mechanical properties. It was found approx. 1% difference. The largest percent change was monitored in the Charpy impact testing, which was nearly a 12% difference. The result was found that there is no difference between the as-received and the 2-h treatment for any of the properties.

There are two types of cryogenic treatment; one low-temperature treatment, also-called "cold treatment", in which the temperatures decreases upto −80 °C at dry ice temperature, and the other one is "Deep Cryogenic Treatment" (DCT), at liquid nitrogen temperature, −196 °C held down for many hours and gradually warmed to the room temperature [13].

Based on the critical analysis of literature it may be concluded that techniques of cryogenic treatment (CT) affect the micro structural and mechanical behavior of the materials. It is observed that cryogenic treatment increases in the homogeneity of microstructure which results in enhanced strength and hardness. At the same time the effect on mechanical properties is also observed depending on the length of time during cryogenic treatment. So more and more investigations are requiring to investigate the mechanical properties and microstructure of aluminium alloy after cryogenic treatment.

In the present work, Al 6082 is treated in various manners such as solution treatment and cryogenic treatment. Effects of each treatment on mechanical properties were studied along with fractography of tensile broken specimen.

2. Treatment profiles

Fig. 1 shows the flow chart of cryogenic treatment generally followed for Al 6082. Different cryogenic treatments which were differentiated by the different cycle temperature profile. Common cryogenic treatment temperature profile was shown in Fig. 2. The different categories of cryogenic treatment are (a) Shallow cryogenic treatment (SCT) will have specimen temperature of -80 °C, (b) Deep cryogenic treatment (DCT) having the cooling temperature of 196 °C, followed by slowly warming to room temperature.

Fig. 2 shows the temperature profile during the cryogenic treatment as a function of time. It is clear that in the cryogenic treatment, temperature reduced to −200 °C in 4 h, hold for 24 h and then temperature reaches to normal room temperature in next 4 h.

3. Material and specimen preparation

As per the various applications of Aluminium alloy Al-6082 is selected for the experiment. It is used in aerospace industry because of its low density and high thermal expansion coefficient [14]. Although Cu is also a material which has higher thermal expansion coefficient compare to Al but it is not use in aerospace industry because of its density is more than Al. The composition of the Al-6082 used is shown in Table 1.The Physical Properties of Aluminium Al6082 are shown in following Table 2.

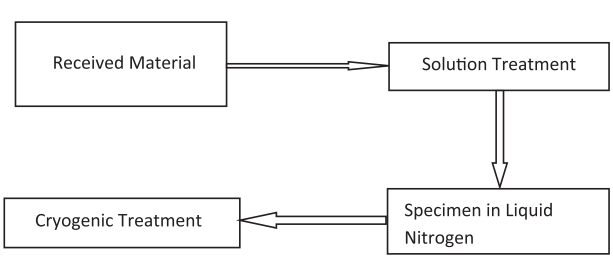

Fig 1. Flow diagram of cyrogenoic treatment for the Al 6082.

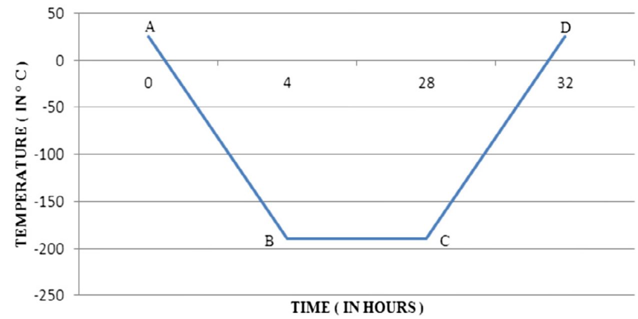

Fig 2. Cryogenic treatment profiles.

4. Experimental layout

A series of experiments has been performed for characterization of treated or not treated specimen. In a hierarchy shown in Fig. 3 explain the complete experimental process.

4.1. Solution treatment

Fig. 4 shows a phase diagram of Al-Mg-Si. Solution heat treatment is carried out at 520 °C for 2 h by heating the alloy into electric furnace so that the a single-phase region followed by rapid cooling. This may increase the hardness and strength of alloy. These alloys may contain smaller amounts of Ti, Fe, Mn, Cr and Zn.

4.2. Cryogenic treatment

Cryogenic treatment of the entire specimen holding it at −183 °C by using liquid nitrogen solution bath for different duration of time 10, 30, 60 min and 24 h were performed and then gradually revert back at room temperature.

5. Mechanical properties evaluation and microstructural characterization

To find the different properties of Al6082 solution and cryogenic treated specimen were evaluated mechanically and microstructural characterization done using optical microscope and SEM. The mechanical characterization was done by evaluating the tensile property and hardness of processed and as received sample.

Tensile Test: For the mechanical properties evaluation, tensile test and hardness test were performed. The tensile test specimen were prepare according to the ASTM: E8-M-08. Testing is to be carried on universal testing machine as shown in Fig. 5.

Hardness Test: Hardness test were performed using Vickers hardness tester, machine consists by a diamond indenter. The full load is normally applied for 10–15 s. The two indentations of diagonal on the surface of the material were indented. After unloading of specimen, diagonal were measured by using a microscope and their average of the diagonal was calculated. The area of the sloping surfaces of the indentation was calculated. The Vickers number (HV) is calculated using the following formula:

Table 1
Composition (wt%) of Al-6082.

Mg	Si	Fe	Cu	Zn	Ti	Mn	Cr	Al
1.20	1.30	0.50	0.10	0.20	0.10	1.0	0.25	Remaining

Table 2
Physical properties of aluminium Al-6082 [7].

S.NO.	Physical Properties	Value
1	Density	2.70 g/cm3
2	Melting Point	555 oC
3	Thermal Expansion	24x10^-6 k
4	Modulus of elasticity	70GPa
5	Thermal Conductivity	180 W/m.k
6	Electrical Resistivity	0.038x10^-6Ω.m

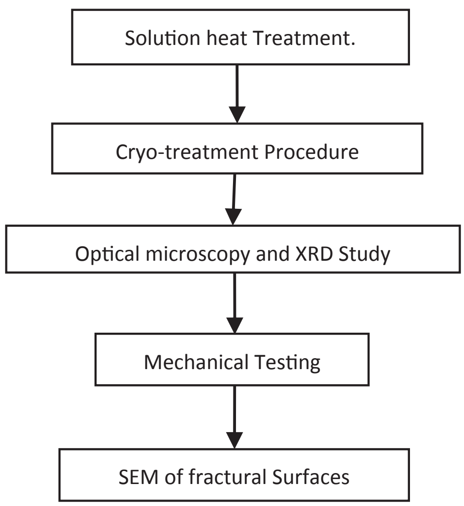

Fig 3. Flow diagram showing complete experimental process.

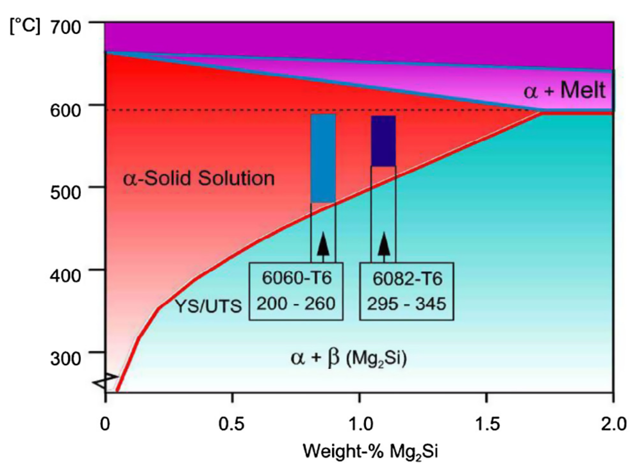

Fig 4. Equilibrium phase diagram Aluminum–Magnesium-Silicon [14].

$$HV = 1.87\left(\frac{f}{d^2}\right) \quad (1)$$

where f & d^2 represent the applied load in kgf and area of the indentation in sq. mm. simultaneously

Microstructural characterization: To understand the effect of treatments on grain size and to capture the fractography, optical microscope and Scanning Electron Microscope (SEM) were used.

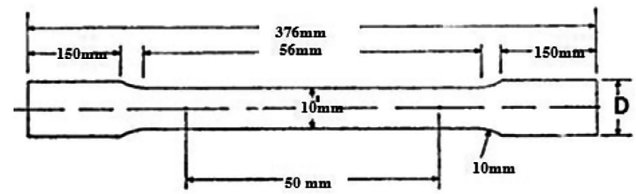

Fig 5. ASTM standard for tensile specimen preparation.

6. Results and discussion

6.1. Tensile test

Tensile testing is to be carried on universal testing machine, according to ASTM: E8-M-08. On specimen of Al6082 solution treated and cryogenic treated with different time zone. Specimen ID notation “ST” and “CT” indicate solution treated cryogenic treated respectively and MT10 indicate material for tensile test after 10 min treatment as shown in Table 3.

Figs. 6 and 7 shows the stress-strain graph of solution treated and cryogenic treated specimen for 10 min respectively. Stress-strain graph of ST and CT specimen for 10 min, 60 min and 24 h were plotted and all the graph indicate the cryo-treatment gives a better result than the solution treatment. Cryo-treatment by increasing the time duration we can observe the increment in ultimate strength.

Figs. 8 and 9 shows the stress-strain graph of cryogenic treated specimen for 60 min and 24 h respectively. It was found that solution treated specimen has the ultimate strength is 270 MPa and after cryogenic treatment for 24 h, it was found to be 348 MPa. This shows that there will be 28.88% increment in ultimate tensile strength of the specimen after cryogenic treatment for 24 h as compared with the solution heat treated specimen. The yields stress of cryogenic treated specimen for 24 h were increased by 350 MPa as compared to the solution treated specimens. To find the causes of strengthening of Al6082 by cryogenic treatment, fractographic analysis by SEM on fracture surface of tensile tested specimen were performed. Fig. 10 shows the comparative bar chart of yield strength and ultimate tensile strength of solution and cryogenic treatment samples.

6.2. Fractographic analysis of tensile specimens

To determine the mechanics of increased strength, the scanning electron microscope images of the different specimen after tensile testing were conducted. Figs. 11 and 12 shows the SEM images of fractured specimen of solution and 24 hrs cryogenic treated samples respectively.

It can be clearly observed from the images that the numbers of pits are more on fractured surface of CT specimen as compared to pits on fractured surface of ST specimen. It observed that there is more surface roughness on fractured surface of CT specimen as compared to fractured surface of ST specimen. Higher roughness value indicates more ductility, thus the cryogenic treatment of Al6082 not only increases the crystalline size but also the ductility of the material increases.

Table 3
Tensile Test Results on the various samples mentioned through specimen ID.

Specimen ID	Elongation (%)	Ultimate Load (KN)	Ultimate Stress (MPa)	Yield stress (MPa)
STMT1	23	21.2	270	10
CTMT10-1	30	25.46	324	52
CTMT60-1	26	25.96	330	51
CTMT24-1	24	27.34	348	45

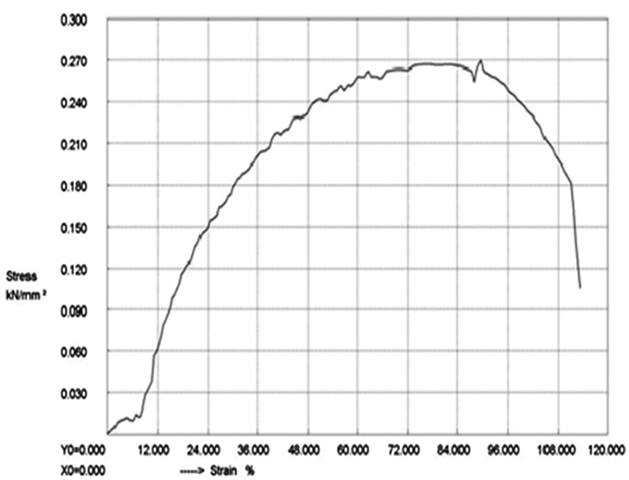

Fig 6. Stress-strain graph of STspecimen.

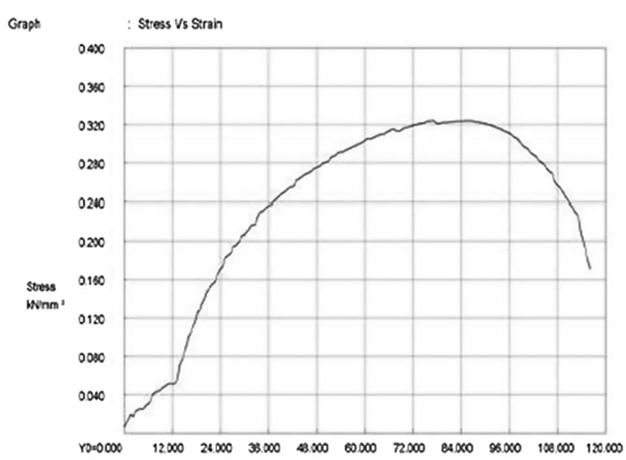

Fig 7. Stress-strain graph of CTspecimen for 10 min.

6.3. Hardness test

Vickers's hardness of CT& ST specimen were prepare and tested by standard vickers's hardness machine. Hardness value of CT& ST are tabulate in Table 4. Result of CT Specimen clearly shows that higher hardness (0.5887 kg/mm^2) as compared to the ST material specimen (0.3473 kg/mm^2). Value of hardness is increasing by increasing the holding time of CT specimen as shown in bar graph in Fig. 14. Increased hardness is attributed to strain hardening. Specimen ID NSHT indicate non solution specimen hardness test and SHT meant to solution treated specimen hardness test same as CTH10 indicate cryogenic treated specimen hardness test for 10 min. Fig. 13 shows the image of the indentation made during the hardness testing. The diagonal of the indent was measured to calculate the hardness of the material.

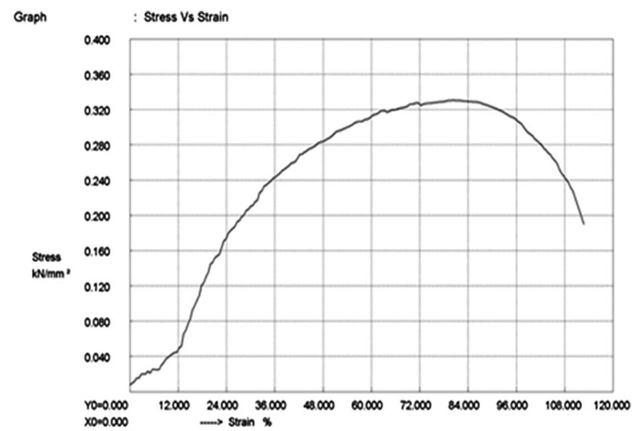

Fig 8. Stress-strain graph of CT specimen for 60 min.

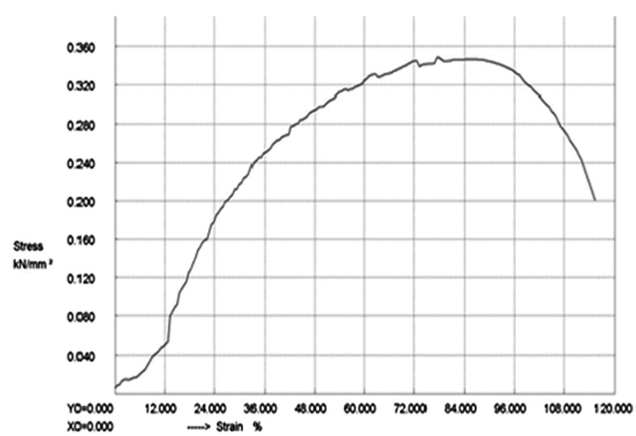

Fig 9. Stress vs. strain graph of CT specimen for 24 h.

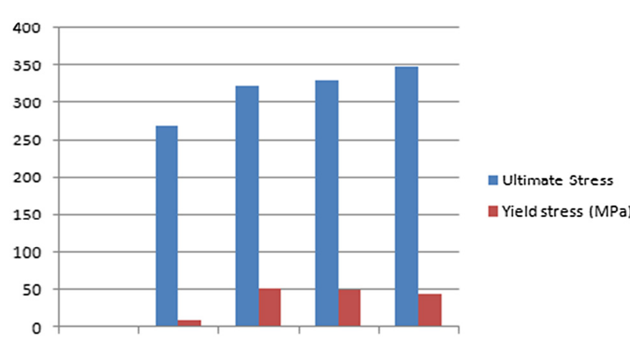

Fig 10. Ultimate stress and yield stress (MPa) of different specimens.

Vickers's hardness of cryogenically treated specimen was 0.5887 kg/mm^2 as compared to the solution treated specimen 0.3473 kg/mm^2. Hardness was 69.5% higher in case of cyro-

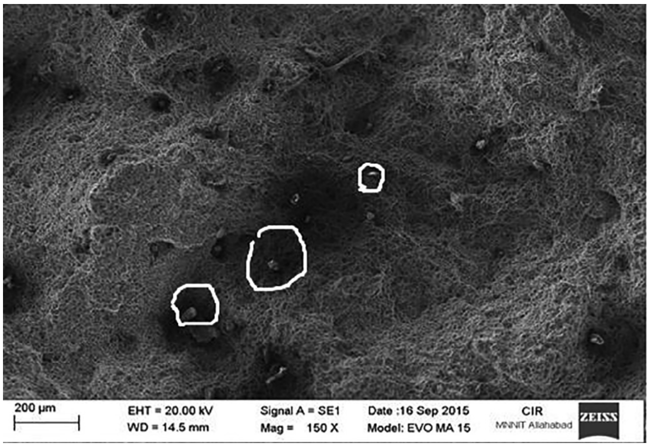

Fig 11. SEM image of STS specimen at 150×.

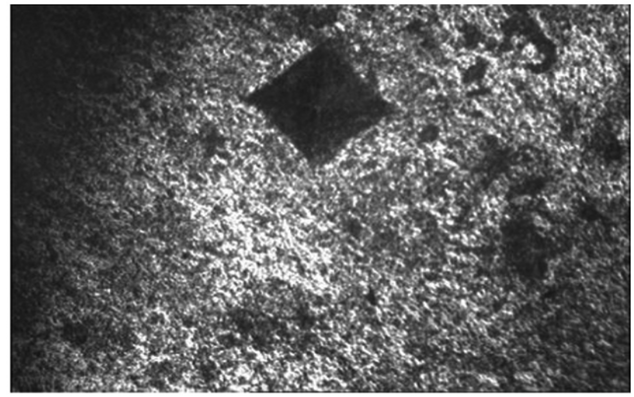

Fig 13. Impression made by indenter on surface of surface of specimen during the hardness test.

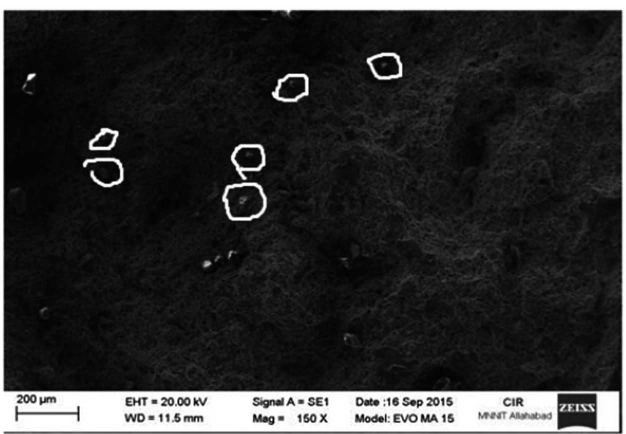

Fig 12. SEM image of CT specimen at 150×.

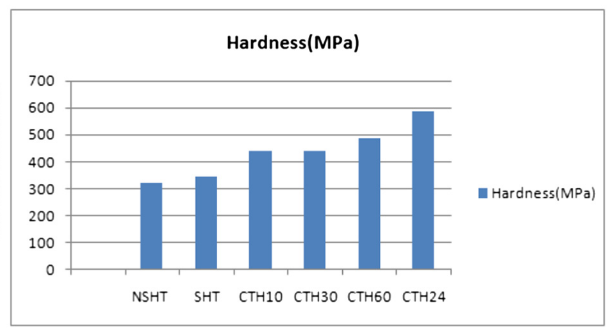

Fig 14. Vickers's hardness of different specimens.

treated specimen as compared to the solution treated specimens. The Increment in the vickers hardness can be attributed due to strain hardening. Fig. 14 shows the bar chart of the vickers hardness of the different samples.

6.4. Optical micrographs

Optical micrograph was performed on 100 µm scale for finding the effect of ST and CTspecimen on the micro structure of Al6082 and shown in Fig. 15a and b.

To understand the effect of cryogenic treatment on microstructure of Al 6082 optical micrographs are shown in Fig. 15. From Fig. 15, it is clear that the grain size decreases on CT samples as compared to ST sample for a same duration of treatments.

7. Conclusion

In present work, a comparative study of solution treatment and cryogenic treatment (10 min to 24 h) on aluminium AA 6082 alloy has been conducted. The effect of solution treatment and cryogenic treatment was evaluated by means of change in microstructure and other mechanical properties. From this work following conclusion were drawn:

Table 4
Hardness test results

Specimen ID	Diagonal D1 (mm)	Diagonal D2 (mm)	Avg. Diagonal, D (mm)	Load (in kgf)	HV (kg/mm^2)	Mean Hardness	Mean Hardness
NSHT 1	13	14	13.5	31.25	0.3206	0.3206	320
NSHT 2	13	14	13.5	31.25	0.3206		
SHT-1	13	14	13.5	31.25	0.3206	0.3473	347
SHT-2	12	13	12.5	31.25	0.374		
CTH10-1	11	12	11.5	31.25	0.4418	0.4418	441
CTH10-2	11	12	11.5	31.25	0.4418		
CTH30-1	11	12	11.5	31.25	0.4418	0.4418	441
CTH30-2	11	12	11.5	31.25	0.4418		
CTH60-1	10	11	10.5	31.25	0.5300	0.4859	485
CTH60-2	11	12	11.5	31.25	0.4418		
CTH24-1	9	10	9.5	31.25	0.6475	0.5887	588
CTH24-2	10	11	10.5	31.25	0.5300		

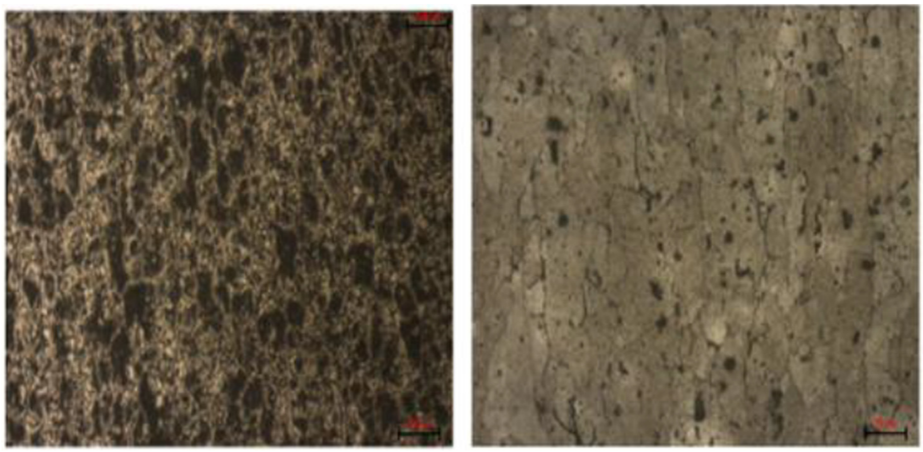

Fig 15. Microstructure of Al 6082specimen after; (a) ST and (b) CT.

(I) Vickers's hardness of cryogenically treated specimen was 0.5887 Kg/mm^2 as compared to the solution treated specimen 0.3473 kg/mm^2. Hardness was 69.5% higher in case of cyro-treated specimen as compared to the solution treated specimens. The Increment in the vickers hardness can be attributed due to strain hardening.

(II) To study the effect of cryogenic treatment on microstructure of Al 6082 optical microscopy is carried out. It is observed in optical microscopy that as compared to solution treated material, the grain size of cryogenic treated material decreases as the duration of cyro- treatment is increased

(III) Tensile strength of cryogenically treated specimen was higher as compared to the solution treated material specimen. The tensile strength of the material increases with increases in cyrogenic treatment time. The maximum increment of about 29% was observed in ultimate tensile strength of 24 h of cyro-treated specimen as compared with the solution treated specimen.

Declaration of competing interests

The authors declare that they have no known competing financialinterestsor personal relationships that could have appeared to influence the work reported in this paper.

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