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Interpretation of hardness of CRYO and RT ECAPed Aluminium 6063 using micro-Raman spectroscopy

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ABSTRACT

The severe plastic deformation technique that has attracted much attention from the material community in present days is Equal Channel Angular Pressing (ECAP). Ultra fine-grained microstructures can be produced by this technique, without a significant change in geometry. Present work compares between Hardness and Raman spectroscopic analysis of CRYO and RT ECAP of Aluminium 6063 alloy .For different aging times different Ramanspectroscopy graphs obtained will be different. The present work purpose is to make use of Raman intensity ratio(I_D/IG) to infer Micro vickers Hardness of CRYO and RT ECAP of Aluminium 6063 alloy.The raman spectroscopy utilizing laser as the light source makes it sutiable for mesurments of different material properties. In this work Raman spectra of CRYO and RT ECAPed Aluminium 6063 at different aging timings at 180^0c temperture will be analysed. These results showed good agreement with age hardening behavior of CRYO and RT ECAPed Aluminum 6063.

Keywords: *Aluminium 6063, Raman Spectroscopy, Ecap*

1.INTRODUCTION

Aluminium 6063 is one of age hardenable Aluminium alloys and widely used in aircraft and automobile industries.These Aluminium alloys applications are increasing day to day because of these strength to weight ratio.ECAP is one of severe plastic deformation technique to modify its structure to nano crystalline scale to improve properties. Equal Channel Angular Pressing (ECAP)is the most used comman process of SPD,which involves pressing a billet through a die consisting of two channels of equal cross sections, intersecting at an angle 110°. The process of ECAP allows us to produce very large plastic deformations to a work-piece without changing the overall dimensions of the work-piece.The ecaped alloy under go through Artificial aging is done by re heating to elevated temperature to dvelop its full strength for different timings. The Material hardness is termed as the opposing ability of the material to localized plastic deformation induced by an indenter with a known load. The word "micro hardness" is defined for micrometer size indentations and low applied loads.Hardness is often used to measure variations in surface properties for surface treatments like shot peening. Hardness is measured on transverse sections of specimen at constant load. For a applied load value, if their is less penetration , the harder is the material. This paper proposes to correlate the hardness of ecaped aluminium 6063 samples age hardened at different timings using micro-Raman spectroscopy, for benchmarking. Therefore, the hardness of the ecaped aluminium 6063 can be infered from raman signals.

2. EXPERIMENTAL DETAILS

2.1 Sampling

Aluminum 6063 commercially available alloy one meter long and 9.5 mm X 9.5 mm square rods are procured. The square rods of Al6063 alloy are cut into samples of 100mm length using abrasive cutter. The curvature is made with the help of a grinding machine. specimens are made to the required dimensions i.e ., 9.5×9.5×100 and the outer curvature is 36^0 .Fig 2.1.1 shows the sample prepared

Fig 2.1.1 Sample Prepared

Solutionizing is done to the samples .The solution treatment is performed at 520^0C.It is carried out in muffle furnance The samples were heated at 520^0C for two hours and quenched in water. During heating the alloy, all the alloying elements will be dissolved to the maximum extent and form a supersaturated solid solution. The quenching treatment after solutionising will retain the supersaturated solution. sample placed in the furnace is shown in Fig 2.1.2

Fig 2.1.2 Samples Placed In The Furnance

2.2 ECAP

Equal channel angular pressing is one of the techniques in metal forming processes in which an ultra-large plastic strain is imposed on a bulk material.ECAP is carried on “Universal Testing Machine”. ecap die with a channel angle of 108 ^{0}and made of high carbon high chromium steel in doing the process with outer curvature angle 36^0 andThe Molybdenum disulfide (MoS_2) used as lubricant . Die used in ecap is shown in Fig 2.2.1

Fig 2.2.1: Die Used For Ecap

While pressing operation is going on, in order to hold the split die, not to get separated clamps are required. The ECAP die used for the experiment was a two-piece split die housed in a square channel with an area of 9.8 x 9.8 mm. The entry channel of 120mm length was made so that 100 mm specimen can be used for the fabrication. After the operation is over, to remove the specimen from the die, care has taken the die design in the split configuration. Lubrication plays any important role during the passage of the specimen, along with smoothness of the die. Molybdenum disulphide (MoS2) is uses as lubricant, in order to free passage of the specimen in the channel. It also helps to minimize the friction on heat during the process.Fig 2.2.2 shows the die setup on the utm

Fig 2.2.2 Die Set Up On Utm

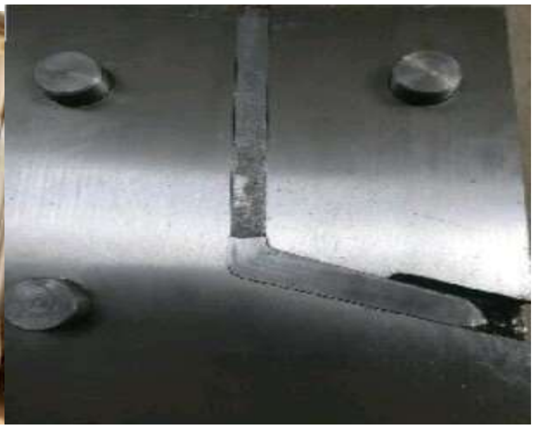


Fig 2.2.3 Sample Extruded In The Die

Initially the ECAP dies were kept on the platform provided on Universal Testing Machine. The lubricant was applied on the die cavity (channel) through which the sample was extruded. Then a tool steel punch was placed on the sample put just into the cavity before the start of the processing. Now the compressor in Universal Testing Machine is lowered down such that the punch and sample assembly sits firmly. Once the assembly is firm then that loads are applied to the punch which forces the samples through the channel and shearing occurs for at the junction connecting the channels gradually the rate of load was increased and at a point a sudden drop of load was observed.. This sudden fall in load during extrusion of the sample is an indication that the sample has passed completely across the arc connecting the two channels. Then the UTM is stopped and the ECAP dies are separated by removing the clamps attached to the ECAP dies. Then the punch and the sample are carefully removed using pointed screw driver with the hammering action. Fig 2.2.3 shows sample extruded in the die But a single pass alone cannot give an ultra fine grain size. sample was again placed into the die cavity by applying lubricant in the channel of die carefully for easy movement of the sample while extruding the sample out this process of filing of the extruded sample is repeated for 4 passes . Fig 2.2.4 shows the Rt ecaped samples

Fig 2.2.4 Ecaped Samples

2.3Age Hardening

After ECAP the specimens were cut into small pieces suitable for the micro Vickers hardness test. The age hardening behaviour was studied only on the middle portion of the samples, after removing the both the ends. In the dimensions 9.5mm×9.5mm×10mm. The ECAPed samples of 4 numbers were subjected to age hardening . Aging is heating the sample below the solvus temperature ie ,. At 180^0 c to produce a finely dispersed precipitate. It is Aged up to 1.5 hours in time intervals of 30 minitus,after every 30 minutes a sample is taken and named according to aging time. This aging process is carreied out in muffle furnance. Then after every 30 minutes the furnace door is opened and with the help of tongs and are water quenched. Then immediately the furnace door is closed and allowed for the aging of other samples, These quenched samples are removed after they cooled and appropriately marked for further differentiation. Hence after respective times the sample are quenched and then marked carefully so that they are properly differentiated. Quenching of the samples are done using water as the media. Figure 2.3.1 shows the furnnace used for aging and water for quenching

Fig 2.3.1Furnance used for aging and quenched in water

2.4 Micro Vickers Hardness Test

For the purpose of hardness test, the samples were polished with various grinding papers of 1/0, 2/0, 3/0 and 4/0. The finer paper causes less surface deformation. Samples were polished thoroughly after each polishing on each emery paper to prevent the crossover of abrasive particles to the next emery paper. To achieve smooth finishing, 4/0 paper is polished using kerosene.

The Vickers micro hardness test is performed using a square-based pyramidal-shaped diamond indenter, leading to a square mark on the surface of sample.The diamond indenter penetrates the sample surface with a known force, $F_{HV,}$ with a dwell time of 10s. The diagonals measured and Vickers hardness is computed as (ASTM E384-17, 2017)

$$HV = 1854.4\ FHV/d2\ HV$$

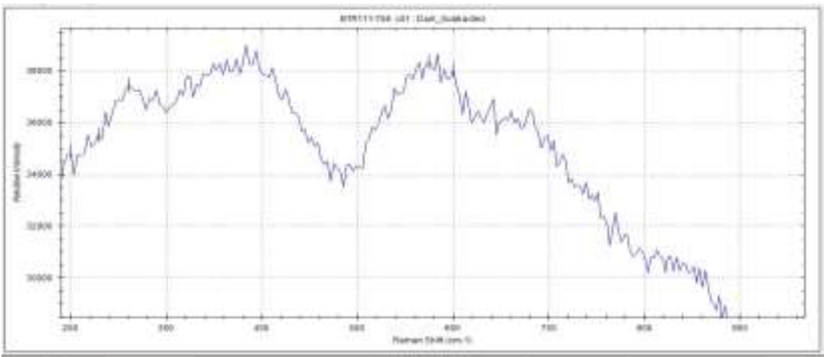
Here F_{HV} is expressed in grams-force (gf) and d is the average of diagonals d1 and d2 which are in micrometers. The gram-force is unit of force and represents the d2 d1 F . The diagonals d1 and d2 are measured after indentation with a applied force F force exerted by a gram of force. The Vickers hardness scale is reported in HV. The indentation is taken at two or three different places and the average of them are tabulated ,this average determines the hardness of the specimen .A load of 300gf is applied. Hardness of ecaped Aluminium 6063 is shown in table2.4

TABLE 2.4 Hardness Of Ecaped Alloy

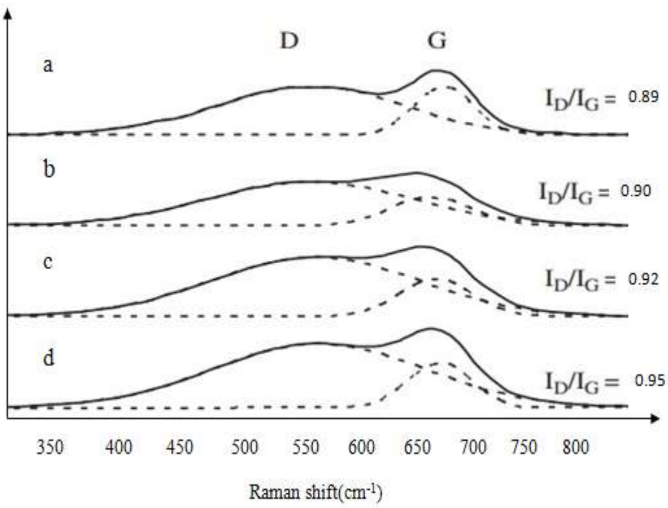
AGING TIME (minitues)	HARDNESS (HV)
0	85.6
30	90.1
60	93.2
90	105.6

2.5 Raman Spectroscopy

This test is carried out on BW-TEK Mini Raman testing equipment. This works on light scatering phenomena. In this the specimen of size 9.5mm×9.5mm×10mm is placed under "Fiber Coupled Raman Trigger Probe".A Raman probe is capable of directing and focusing the monochromatic excitation source (typically a laser) to the sample, collecting the scattered light and then directing it to the spectrometer. This change in scattered light intensity is termed as raman shift, The generated data is normalized and peak fitting is done and Raman spectrum produce two peaks namely D-peak and G- Peak ,the higest peak produced in the spectrum is G- peak and the preciding peak is D-peak,the ratio of raman shfit intensity values of G peak and D peak are taken as I_D/I_G .In this the intensity ratios are interfered with hardess values with respect to aging time at 180^0c. The generated data is normalized and peak fitting is done



Graph 2.5 .1 Raman Spectrum Generated For 0 Minitues Aging Time



Graph 2.5.2: Raman spectra of ECAPed AL6063 Aged at 180^0c for (a) 0 minitues, (b) 30 minitues, (c) 60 minitues,(d) 90 minitues

Table 2.5 Hardness And Raman Intensity Ratio

AGING TIME (minitues)	HARDNESS (HV)	Id/Ig
0	85.6	0.89
30	90.1	0.90
60	93.2	0.92
90	105.6	0.95

The hardness against the Raman intensity ratio I_D/I_G. The table 2.5 shows that the hardness increases directly with the I_D/I_G ratio. The relationship between the hardness and the Raman intensity ratio I_D/I_G of these samples can be taken as a reference from which the hardness can be determined

3. CONCLUSIONS

As a non-destructive method of characterization of molecular species present on the surface of metals, Raman spectroscopy has many advantages. In this research Raman spectra of ECAPed Aluminium 6063 at different aging time are analysed. Raman intensity ratio(ID/IG) to infer hardness of ECAP of Aluminium 6063 alloy is done. The values of the hardness are increasing with aging time .The hardness increases directly with the ID/IG ratio. In this Raman intensity ratios shows direct increase in intensity ratios along with hardness with change in aging time The relationship between the hardness and the Raman intensity ratio ID/IG of these samples can be taken as a reference from which the hardness of the ecaped AL 6063 can be estimated based on its measured Raman intensity ratio.

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