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Influence of Deep Cryogenic Treatment on the Mechanical Properties of AISI 440C Bearing Steel

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Abstract

This research paper focuses on the influence of shallow and deep cryogenic treatment on the mechanical properties of AISI 440C bearing steel. Hardness of this material is increased by 7% when subjected to Deep Cryogenic Treatment (DCT); and by 4% for Shallow Cryogenic Treatment (SCT) compared to the Conventional Heat Treatment (CHT). 'ImageJ' software has been used to analyse the optical microscope image of the conventional and cryogenic treated samples to estimate the retained austenite percentage. The retained austenite percentages of these samples were 29%, 8% and 5.7% for the conventional, shallow cryogenic and deep cryogenic treated samples respectively. DCT sample shows relatively higher hardness than CHT samples due to higher conversion of retained austenite to martensitic structure. DCT sample shows minor increment in hardness when compared to SCT. DCT and SCT samples show improved hardness by 7% and 4% than that of the CHT sample. Fractography analysis of the cryogenically and conventionally heat treated bearing steel were performed using Scanning Electron Microscope (SEM). SEM analysis clearly indicates the formation of flat facets in SCT specimens and micro cracks in DCT specimens.

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Keywords: AISI 440C Bearing Steel; Conventional Heat Treatment (CHT); Shallow Cryogenic Treatment (SCT); Deep Cryogenic Treatment (DCT); Hardness; Microstructure; Fractography; Retained Austenite.

1. Introduction

AISI 440C bearing steel is a martensitic stainless steel which has good corrosion resistance properties. This steel is termed as high carbon and high chromium steel and is widely used in many engineering components such as ball

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bearings, cutleries, valve parts, ball valves, needle valves, surgical equipments and chisels etc. AISI 440C bearing steel retains its properties even at elevated temperature; hence it is suitable for components operating at high temperature. Normally hardening of steel is carried out as follows; heating the steel to austenitizing temperature and quenching or rapid cooling, which transforms some or all of the soft austenite structure into the higher strength martensitic structure with supersaturated carbon [1]. Hardness can be increased by transforming austenite to martensite. The transformation from austenite to martensite begins and finishes at a well-defined temperature called martensite start temperature (M_S) and martensite finish temperature (M_F). Increasing the carbon content in steels, lowers the martensite's start and finish temperatures, even goes down to sub-zero temperature. As a result, complete transformation of austenite into martensite could not be occurred in conventionally heat treated steel [2]. This results in some percentage of retained austenite in the microstructure of steel. Hence high carbon content AISI 440C steel requires cold heat treatment process to transform most of the retained austenite to martensite.

Cold or cryogenic heat treatment is an additional step in the conventional heat treatment hardening process (CHT). Cryogenic treatment is normally carried out after the conventional heat treatment process is over but before tempering process starts [3]. In this paper, two types of cryogenic treatments were employed, one is shallow cryogenic treatment (SCT) in which the conventionally heat treated (CHT) bearing steel was cooled in freezing chamber. This freezing chamber was maintained at -80°C and was soaked for 5 hours. It is then allowed to reach room temperature. The second method is deep cryogenic treatment (DCT) in which the conventionally heat treated (CHT) samples were subjected to cool down slowly from room temperature to -196°C for 3 hours. It is then soaked for 24 hours and then allowed to reach room temperature. Cryogenic treatment process is carried out in the liquid nitrogen (LN_2) chamber which was well insulated around it. The complete set up of the cryogenic treatment is explained by Das et al. [4]. Finally all the samples were subjected to tempering process at 200°C .

2. Literature Survey

Paulin [5] reported the cryogenic treatment of steel in which some of the retained austenite is converted into martensite and it is an extension of CHT process. It is also noteworthy that cryogenic treatment process significantly improves the life of bearing steels. Lipson [6] elucidated that cryogenic treatment produced smaller grain size in the range of 1-4% in microstructure, and this refinement of grain structure resulted in improving wear resistance. El Mehtedi et al. [7] optimized the process route through deep cryogenic treatment on X30CrMoN 15 1 steel in which improved its hardness and microstructure. Srisiva et al. [8] mentioned that wear resistance improved by 37% for the conventionally heat treated 100Cr6 bearing steel when subjected to DCT. Arockiya Jaswin et al. [9] reported the wear resistance improvement of X45Cr9Si3 and X53Cr22Mn9Ni4N valve steels by cryogenic treatment. They reported that wear resistance of cryogenically treated both valve steels increased by 46.51% and 27.8% respectively than CHT. Sendhilkumar et al. [10] explained the procedure of cryogenic treatment on En 19 steel and reported that wear resistance noticeably increased by 118.38% for SCT samples and 214.94% for DCT samples when compared to CHT samples. In addition, wear resistance increased for the DCT was 44.39% with respect to SCT samples due to possible transformation some of the retained austenite to martensite. Mahdi Koneshlou et al. [11] found that cryogenic treatment of AISI H13 hot work tool steel is induced precipitation of more and uniform carbide particles thereby increased wear resistance.

Shaohong Li et al. [12] and Cajner F. et al. [13] explained that cryogenic treatment on the die steel results in decreased toughness in consequence with increased hardness. Podgornik et al. [14] studied the tribological properties of DCT specimens over of P/M high speed steel. DCT exhibited higher abrasive wear resistance and better galling properties. Joseph Vimal et al. [15] elucidated that improvement of wear resistance of En 31 steel subjected DCT attributed to transformation of retained austenite to martensite and precipitation of eta-carbide particles. Kalin et al. [16] showed experimental results in which wear resistance behavior of cryogenic treated high speed steels at various loads was of higher order magnitude than vacuum heat treated. Zhirafar et al. [17] presented mechanical properties of cryogenic treated 4030 steel. Hardness is increased with results of conversion of retained austenite to martensite. Huang et al. [18] studied the effect of DCT on microstructure of M2 tool steel. Hardness improvement is attributed to the increase of carbide density in microstructure due to carbon clustering.

3. Experimental procedure

In this study, AISI 440C bearing steel of 10x10 cm square cross section was subjected to CHT, SCT and DCT. The composition of the steel was determined using Optical Emission Spectroscopy (OES) and is presented in table 1. Typical cryogenic heat treatment process is shown in figure 1. Samples were subjected to CHT as per ASM standards [1] followed by hardening and quenching. SCT and DCT were carried out after tempering process at 200°C. These samples were subjected to SCT as explained by Harish et al [2]. During SCT, the samples were placed in an insulated freezer at -80°C and then soaked for 5 hours for attaining the steady state temperature then allowed to reach the room temperature. In the case of DCT, samples were cooled down slowly to very low temperature -196°C within 3 hours. Then samples were soaked at the same temperature of -196°C for 24 hours. Finally the samples were slowly heated back to room temperature for 6 hours. Hardness test is carried out on Rockwell hardness testing machine for CHT, SCT and DCT samples as per standard procedures. The table 5 shows hardness test results.

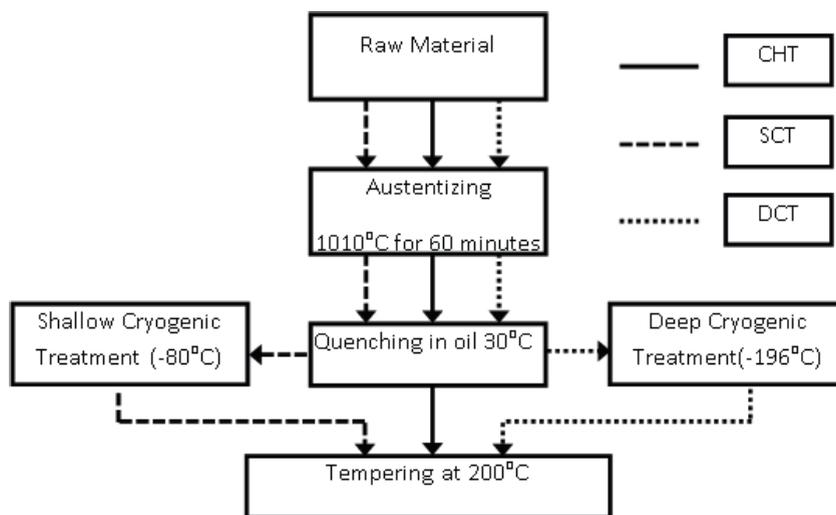


Fig.1. Cryogenic heat treating process

Table 1 Chemical analysis of AISI 440C raw material

Element	Wt. Percentage
Carbon(C)	0.93
Chromium(Cr)	16.94
Manganese(Mn)	0.40
Silicon(Si)	0.74
Molybdenum(Mo)	0.45
Iron(Fe)	Remaining

3.1. Charpy impact test

The conventionally and cryogenically treated samples were subjected to Charpy impact test and samples were prepared as per ASM standards. The impact force is measured by attaching strain gauges on the striker. Table 6 shows the Impact test results.

3.2. Microstructural study

The microstructures of cryogenic and conventionally treated samples were analyzed by Optical Microscopy (OM). Samples were polished using SiC emery papers grades as follows 600, 800 and 1000 and then super finished using diamond paste on the velvet cloth disc with kerosene as the suspension medium. Etching of these samples were carried out using 2% nital (nitric acid and ethanol) and later they were washed with acetone and then dried in air. Microstructures of samples were revealed at a magnification of 500X. Then the micrographs of conventional and cryogenic treated samples were analyzed through the 'ImageJ' software. Renee Heibronner et al. [19], Sumin Zhu et al. [20] and Sitarama Raju et al. [21] were used 'ImageJ' software as an analyzing tool for the estimation of grain size and grain shape from the optical microscope image.

4. Result and Discussion

4.1. Microstructural analysis

Micrograph and analysed micrograph of conventionally heat treated sample are shown in the following figures 2 and 3. The analysed micrograph data of CHT sample is presented in table 2. From the figure 2, the observation of white regions in the microstructure indicates retained austenite. This figure also shows the low level formation of carbide particles due to poor precipitation. From the observation of table 2, the level of retained austenite of CHT sample is 29%.

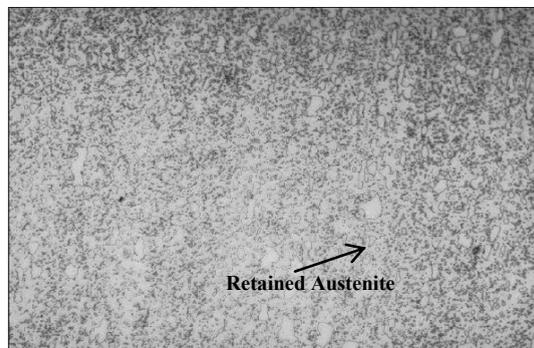


Fig. 2 Micrograph of CHT sample

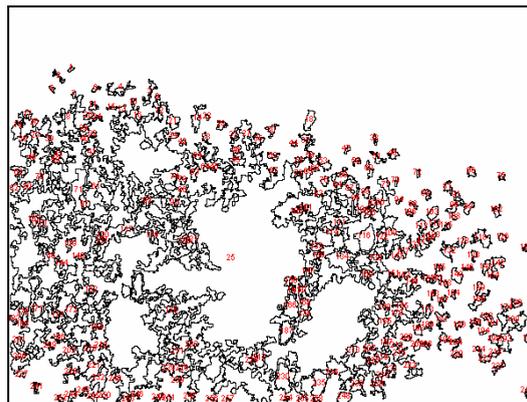


Fig. 3 Analyzed micrograph of CHT sample

Table 2 Analyzed data of micrograph of CHT sample

Slice	Count	Total Area	Average S	% Area	Mean
Microstructure	258	79303	307.376	29.404	10.958

Micrograph and analyzed micrograph of shallow cryogenic treated sample are shown in the following figures 4 and 5. The analysed micrograph data of SCT sample is presented in table 3. The microstructure indicates the presence of martensite at tempered condition and precipitated carbide particles with significant level of retained austenite. From the observation of table 3 the level of retained austenite is about 8% for SCT sample.

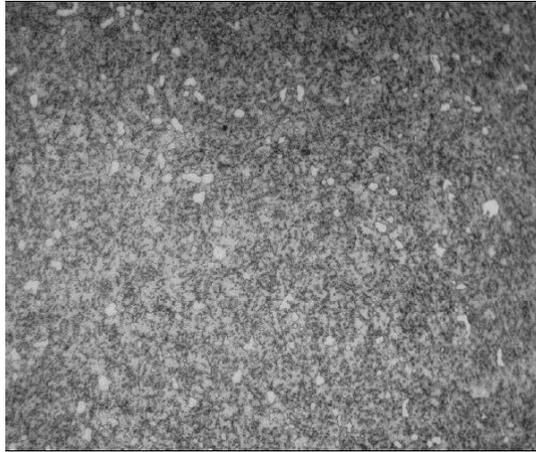


Fig. 4 Micrograph of SCT sample

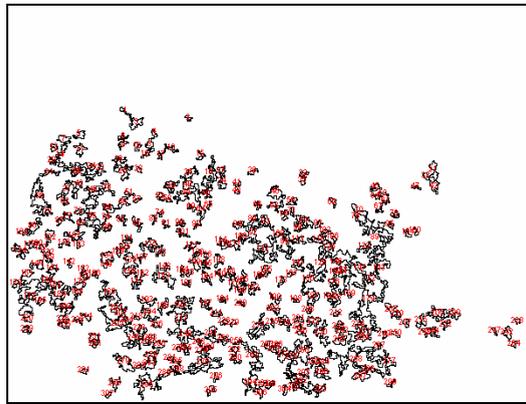


Fig. 5 Analyzed micrograph of SCT sample

Table 3 Analyzed data of micrograph of SCT sample

Slice	Count	Total Area	Average S	% Area	Mean
Microstructure	307	21947	71.489	8.106	247.569

Micrograph and analysed micrograph of deep cryogenic treated sample are shown in the following figures 6 and 7. The analysed micrograph data of DCT sample is presented in table 4. This microstructure shows larger amount of martensite at tempered condition with finely dispersed precipitated carbide particles. From the observation of table 4 the level of retained austenite is about 5.7% for DCT sample. It is understood from the figures 3, 5 and 7 that the presence of retained austenite decreased as a result of cryogenic heat treatment.

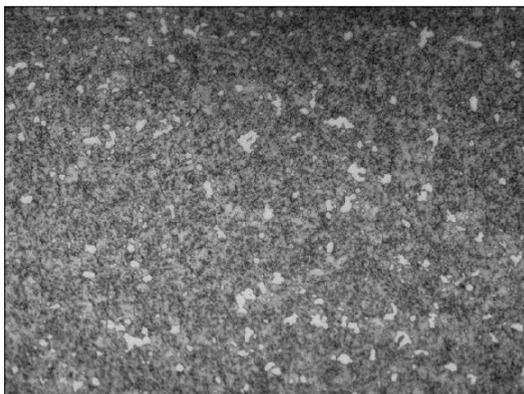


Fig. 6 Micrograph of DCT sample

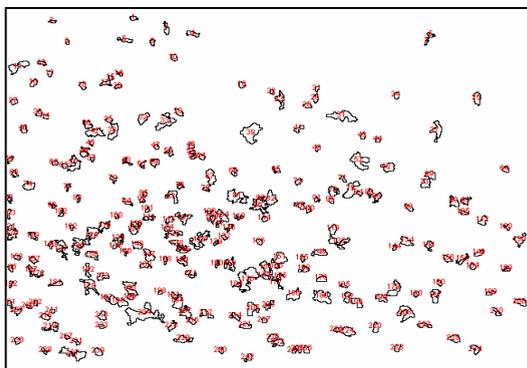


Fig. 7 Analyzed micrograph of DCT sample

Table 4 Analyzed data of micrograph of DCT sample

Slice	Count	Total Area	Average S	% Area	Mean
Microstructure	242	14988	61.934	5.736	1.527

4.2. Hardness Test results

The hardness values for the conventional, shallow and deep cryogenic treated samples are presented in table 5. From the results it is clear that the conventionally treated AISI 440C bearing steel has lower hardness compared to shallow and deep cryogenic treated samples. The DCT samples were of highest hardness of SCT and CHT samples due to higher conversion of retained austenite to martensitic structure than SCT and CHT samples. However, DCT sample shows only comparable increase in hardness than SCT. DCT and SCT samples show improved hardness by 7% and 4% than that of the CHT sample.

Table 5 Result of Hardness Test

Sample	Rockwell hardness HRC
Raw Material	20
CHT	57
SCT	59
DCT	61

4.3. Impact test

The impact energy of the conventional, shallow cryogenic and deep cryogenic treated samples are comparable as shown in table 6. It is understood that low impact value indicates lowest energy absorption by the material. It leads to brittle failure and also reduction in toughness. Table 6 directly means that there is no remarkable change in toughness due to the shallow and deep cryogenic treatments.

Table 6 Impact energy of conventional, shallow and deep cryogenic treated samples

Sample	Impact Energy(J)
CHT	2.4
SCT	2.2
DCT	2.0

4.4. Fractograph analysis

After the impact test, the fractured surfaces of conventional, shallow cryogenic and deep cryogenic treated samples were analyzed using Scanning Electron Microscope (SEM). The SEM images of tempered conventional, shallow cryogenic and deep cryogenic treated samples at the magnification of 5000X are shown in Fig 8-10.

Figure 8 shows the fractograph of CHT specimen. The fractograph of the fractured surface consists of cleavage facets and the small facets appearing along the cleavage plane. Carbide particles have promoted brittle mode of fracture.

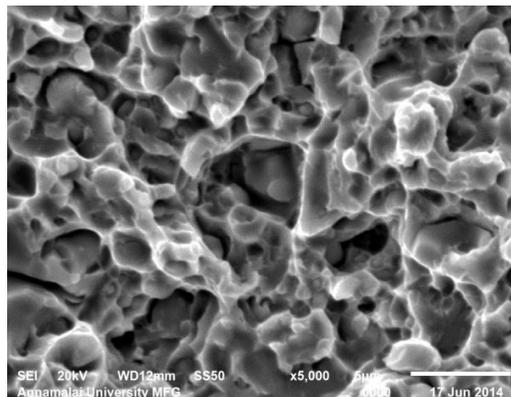


Fig. 8 Fractograph of CHT sample

Figure 9 shows the fractograph of SCT specimen in which flat facets of cleavage are seen in the region of fracture. This figure shows a predominantly cleavage rupture due to fine carbide particles formed during the cryogenic treatment.

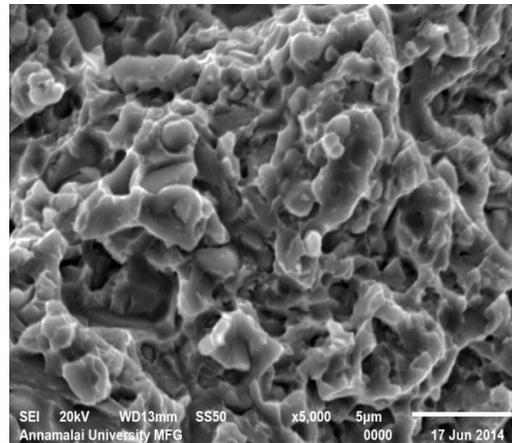


Fig. 9 Fractograph of SCT sample

Figure 10 shows the fractograph of deep cryogenic treated specimen. Very fine sized carbide particles leading to local cleavages were visible in martensitic regions. Deep micro cracks appeared on the interface of austenite to martensitic structures.

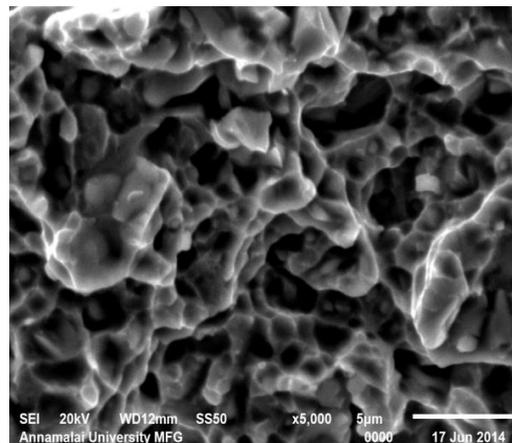


Fig. 10 Fractograph of DCT sample

5. Conclusion

The effect of deep cryogenic treatment (DCT) and shallow cryogenic treatment (SCT) on AISI 440C bearing steel were analyzed. Hardness of deep and shallow cryogenic treated samples was increased by 7% and 4% respectively to that of CHT specimen. This is due to higher percentage of conversion of retained austenite to martensite in DCT and SCT than CHT. The impact energy for the conventional, shallow cryogenic and deep cryogenic treated samples were comparable and there is minor difference in impact energies and the mode of fracture was brittle for all the specimens. Microstructural analysis shows that CHT specimen has more amount of retained austenite and lesser amount of martensite than deep cryogenic treated (DCT) and shallow cryogenic treated

(SCT) specimens. In deep cryogenic treated specimens, there are less amount of retained austenite than CHT and SCT. Fractograph analysis elucidated that facet cleavage with brittle fracture in CHT and cryogenically heat treated AISI 440C steel.

References

- [1] ASM Handbook , 1991.Heat Treating.
- [2] S. Harish, A. Bensely, D. Mohan Lal, A. RaJadurai, Gyöngyvér B. Lenkey, 2009. Microstructural study of cryogenically treated En 31 bearing steel, *Journal of Materials Processing Technology*, Vol-209 pg: 3351–3357.
- [3] MooreK. Collins.D.N. 1993. Cryogenic treatment of three heat treated tool steels, *Journal of Key Engineering Materials*, Vol-86-87 pg: 47-54.
- [4] D. Das, A.K. Dutta, K.K. Ray, 2008. On the enhancement of wear resistance of toolsteels by cryogenic treatment, *Journal Philosophical Magazine Letters*, Vol- 88 pg: 801–811.
- [5] Paulin P. March 1993. Frozen gears, *Journal Gear Manufacturing*, pg: 26-29.
- [6] Lipson C. 1967. *Wear Consideration in Design*, Prentice-Hall, Engle Wood Cliffs, NJ.
- [7] M. El Mehtedi, P.Ricci, L.Drudi, S.El Mohtadi, M.Cabibbo, S.Spigarcelli, 2012. Analysis of the Effect of Cryogenic Treatment on the hardness and microstructure of X30 CrMoN 15 1 steel, *Journal Materials and Design*, Vol- 33 pg: 136-144.
- [8] Sri Siva R. M. Arockia Jaswin D. Mohan Lal, 2012. Enhancing the Wear Resistance of 100Cr6 Bearing Steel Using Cryogenic Treatment, *Journal Tribology Transactions*, Vol-55 pg: 387-393.
- [9] M.Arockia Jaswin, D. Mohan Lal, A. RaJadurai, 2011. Effect of Cryogenic treatment on the microstructure and wear resistance of X45Cr9Si3 and X53Cr22Mn9Ni4N valve steels, *Journal Tribology Transactions*, Vol-54 pg: 341-350.
- [10] D.Sendhilkumar, I.RaJendran, 2011. Influence of shallow and deep cryogenic treatment on Tribological behaviour of En 19 steel, *Journal of Iron and steel research*, Vol-18(9) pg: 53-59.
- [11] Mahdi Koneshlou , Kaveh Meshinchi Asl , Farzad Khomamizadeh, 2011. Effect of cryogenic treatment on microstructure, mechanical and wear behaviors of AISI H13 hot work tool steel, *Journal Cryogenics*, Vol-51 pg: 55-61.
- [12] Shaohong Li, Yinzi Xie, Xiaochun Wu, 2010. Hardness and toughness investigations of deep cryogenic treated cold work die steel, *Journal Cryogenics*, Vol-50 pg: 89-92.
- [13] FranJo CaJner, VoJteh Leskovsek, Darko Landek, HrvoJe CaJner, 2009. Effect of deep cryogenic treatment on high speed steel properties, *Journal Materials and Manufacturing processes*, Vol-24 pg: 743-746.
- [14] B. Podgornik, V. Leskovsek, and J.Vizintin, 2009. Influence of deep cryogenic treatment on tribological properties of P/M high-speed steel, *Journal Materials and Manufacturing Processes*, Vol-24 pg: 734–738.
- [15] A. Joseph Vimal, A. Bensely, D. Mohan Lal K. Srinivasan, 2008. Deep cryogenic treatment improve the wear resistance of En 31 Steel, *Journal Materials and Manufacturing Process*, Vol-A339 pg: 369-376.
- [16] M. Kalin, V. Leskovsek J.Vizintin, 2006. Wear behaviour of deep cryogenic treated high speed steel at different loads, *Journal Materials and Manufacturing Process*, Vol-21 pg: 741-746.
- [17] S. Zhirafar, A. Rezaeian b, M. Pugh, 2007. Effect of cryogenic treatment on the mechanical properties of 4340 steel, *Journal of Materials Processing Technology*, Vol-186 pg: 298–303.
- [18] J.Y.Huang. Y. T Zhu, X.Z Liao, I. J. Beyerlein, M.A Bourke, T. E Mitchell, 2003. Microstructure of cryogenic treated M2 tool steel, *Journal Material Science and Engineering Vol-A339* pg: 241-244.
- [19] Raneë Heilbronner, Nynke Keulen, 2006. Grain size and grain shape analysis of fault rocks *Journal Tectonophysics*, Vol-427 pg: 199-216.
- [20] Sumin Zhu, William G. Fahernholtz, Gregory E. Hilmas, 2007. Influence of silicon carbide prticle size on the microstructure and mechanical properties of zirconium diboride-silicon carbide ceramics, *Journal of the European Ceramic Society*, Vol-27 pg: 2077-2083.
- [21] K. Sitarama RaJu, M.Ghanashyam krishna, K.A.Padmanabhan, K.Muraleedharan N.P, 2008. Grain size and grain boundary character distribution in ultra-fine grained (ECAP) nickel, *Journal Material science and engineering*, Vol-491 pg: 1-7.