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Controlled Cryo Cooling of Hard Engineering Materials Affecting Mechanical Properties

Raghu yogaraju[a], P. Sampath Kumaran[b], S. Seetharamu[b], M. Venkatarama Reddy[a], D.S. Nadig[c]

[a] *Department of Mechanical Engineering, Bangalore Institute of Technology, Bangalore, India*
[b] *Materials Technology Division, Central Power Research Institute, Bangalore, India*
[c] *Centre for cryogenic treatment, Indian Institute of Science, Bangalore, India*

ABSTRACT

The success of any component material is depending on its workability and durability. The present work is focused on improvement of mechanical properties with a supplement to the conventional heat treatment. The cryogenic treatment is the alleviate process for improving the mechanical properties. The effect of cryogenic treatment on the retained austenite, hardness, residual stress and microstructure of some selected engineering materials is studied. The selected samples are subjected to Cryogenic treatment in comparison with the untreated ones have been investigated and the cryogenic treated specimens have shown improvement in the in terms of retained austenite getting transformed to tempered martensite and other with increase in hardness, reliving of residual stress. The X-ray diffraction (XRD) technique reveals the level of martensite and austenite, the microstructure features method shown relative results

Keywords – Martensite, austenite, XRD, Cryogenic treatment.

1. INTRODUCTION

The materials that are suitable under compressive and tensile loading find its application in various fields from cutting tool industries to power plants. The engineering materials tend to degrade due to service conditions and in such situations one look for improved properties to enhance feasible life cycle by adopting suitable processes such as heat treatment, tempering, cooling procedures.

The present work focuses on the controlled cryogenic treatment to enhance the mechanical and metallurgical characteristics of engineering materials. The cryogenic treatment may be simplified into a process of chilling a part down to relatively near absolute zero and maintaining that condition until the material has cold-soaked. Exposing the metal tools, machine and other wearing parts to a very low temperature leads to transformation in the microstructure, usually in ferrous materials.The term controlled cryo-treatment refers to the process to be carried out in a methodical way, wherein the material is cooled slowly to the cryogenic temperature at a specified rate and soaked at that temperature for a long duration of time. Then it is brought back to the room temperature gradually. The results of cryo-treatment on cutting tools especially steels have been reported and it is encouraging, but still there is a large scope of improvement from the point of studying the effect of cryogenic treatment on the dimensional stability, increase in hardness, removal of residual stresses, conversion of retained austenite to martensite etc.

Hardness is a characteristic of a solid material and offers resistance to permanent deformation and expressed in various scales such as Rockwell, Vickers, and Brinell etc. It is known that that the hardness increases with decrease in practical/grain size. The other parameter viz., residual stresses are the locked up stresses in a component, which arise due to production processes involving forming, rolling, welding, grinding, heat treatment and certain fabrication methods on complicated structures leading into formation of stress. The influence of stresses on the premature failure of metallic structures has very well been recognized. Such stresses critically control the durability and functionality requirements. An understanding of residual stress allows the designers to predict the possible modes of damage that occur and hence predict the performance and life of the component.

Hardening of steels requires that the material be heated to a high temperature followed by a quenching and tempering process. During the heating cycle, the room temperature phase is transformed into a face-centered cubic structure known as Austenite. During quenching, the Austenite will then transform into fresh Martensite, which is a very hard, but brittle phase. Thus, a tempering process is almost always undertaken to reduce the brittleness of the steel at the expense of a slight loss in hardness. In real life, however, the heat treatment process is not as ideal as this. Often, some of the Austenite will be retained after quenching and tempering, which can lead to degradation in the material's performance. This is due to the fact that the retained Austenite can be transformed into fresh, untempered Martensite by applied stresses while in use. Also, the transformation of the retained Austenite will cause a dimensional instability in the part, leading to QC problems. One final concern with high carbon steels is that some of the carbide phases do not dissolve completely during the Austenitizing treatment. This will lead to complications in the determination of the amount of retained Austenite and will require careful analysis.

2. EXPERIMENTAL

2.1. CRYOGENIC TREATMENT

The cryo-treatment system incorporates mainly the cryo-treatment chamber and an auxiliary LN2 supply system to supply controlled quantity of LN2 to the chamber to maintain the rate of cooling, soaking and warm up. The controlled LN2 supply to the chamber is carried out by using a solenoid valve activated by a PID controller shown in figure 1.

The cryogenic treatment unit shown in figure 2 is made of stainless steel with polyurethane foam (PUF) insulation. It has a fan-motor assembly mounted centrally in the top cover. The samples housed in the meshed trays are gradually cooled to cryogenic temperature by the cold nitrogen gas forced convection continuously produced by the rotating fan housed below the buffer tank containing LN2 and the cold exchange from LN2 circulating in the copper tube heat exchanger brazed on the outer wall of the metallic shroud housed around the meshed trays. The LN2 supply is regulated by a solenoid valve operated by a PID controller with predetermined set points. Various cryo-treatment cycles can be programmed to suit the requirements. The temperatures of the specimens are measured using Platinum Resistance Temperature Detectors (RTD). The temperature data of the samples read by PID over the period of the cryo-treatment cycle are stored continuously using a data acquisition system.

The samples were cryo treated for a total duration of 45 hours, which include 9 hours of cooling 24 hours of soaking and 12 hours of warming to room temperature. The PID controller was programed as per the present conditions of temperature of 98 k for 24 hours and specimens were allowed to warm up to ambient temperature by closing LN2 supply.

Fig 1: PID controller

Fig 2: Cryo Treatment Setup

2.2. RESIDUAL STRESS AND RETAINED AUSTENITE

In the case of scattering of X-Rays by the crystalline substances, contrary to that by amorphous substances, scattered X-rays due to each atom that composes a crystal are reinforced in a certain specific direction of an extremely narrow angular range and are propagated. This phenomenon is referred to as diffraction. The diffraction angle made by the incident X-Ray beam and a diffracted X-ray determines the retained austenite and residual stress.
The following can be generally know pertaining X-Ray diffraction.

1. By knowing the diffraction angle, it is possible to identify unknown substance or those contained in other substance. Especially, precision measurement of diffraction angle can clarify residual stresses (and accordingly residual strains) that exists in metals.
2. By knowing the intensity of diffracted rays, it is possible to learn the amount of components of matter. (Quantitative analysis. The measurement of retained austenite is based on this principle).

Fig 3: XRD Setup

2.3. HARDNESS

The Rockwell Hardness test is a hardness measurement based on the net increase in depth of impression as a load is applied. In the Rockwell method of hardness testing, the depth of penetration of an indenter under certain arbitrary test conditions is determined.
There are several hardness scales, for experimentation the ‘C’ scale is used with the load of 150 Kgf. The indenter used is a spherical diamond-tipped cone of 120° angle and 0.2 mm tip radius, called Brale. The type of indenter and the test load determine the hardness scale.

2.4. MICRO STRUCTURE STUDY

Microstructure is defined as the structure of a prepared surface or thin foil of material as revealed by a microscope above 25X magnification. The microstructure of a material 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.

3. RESULT AND DISCUSTION

3.1. RETAINED AUSTENITE (RA)

The test results for RA % of selected materials before and after cryo treatment are shown in the table 1. From the result it is observed that there is significant decrement in RA, followed by the treatment and the statements is supported by microstructure and reduce in residual stress.

Sl. No.	Sample Identification	Retained Austenite	
		Before Cryo treatment	After Cryo treatment
1	Spring steel	1.8-2	0.6
2	Hot die steel	1.4-1.6	0.6-0.8
3	High chromium	9.8-10.6	8.3-8.7
4	Nihard 4	12.2-12.9	9.6-10.1
5	EN24	2.3-2.7	0.5-0.6

Table 1: Retained Austenite of samples before and after treatment

3.2. RESIDUAL STRESS

The samples before cryo treatment have shown compressive residual stress which induced during casting or machining is tending to relive, this behavior is mainly due to increased precipitation of fine carbides during treatment.

Sl. No	Sample Identification	Residual Stress (MPa)	
		Before cryo treatment	After cryo treatment
1	Spring steel	-239.46	-115.045
2	Hot die steel	-305.45	- 248.41
3	High chromium	-306.97	-233.612
4	Nihard 4	-516.403	-381.892

Table 2: Residual Stress of samples before and after treatment

Due to changes in austenitic to martensite after treatment, there will be a change in phase from face centered to body centered structure. Due to phase transformation, the compressive stresses are reducing.

3.4. HARDNESS

The hardness test is performed on all the samples before and after the cryogenic treatment using Rockwell hardness tester. The hardness measured is interns of HRC.

Sl. No	SAMPLE	Hardness (HRc) before cryo treatment	Hardness (HRc) after cryo treatment
1	Spring Steel	57	61
2	Hot Die Steel	31	34
3	High Chromium	45	52
4	Nihard-4	47	55
5	EN24	42	44

Table 3: Hardness of samples before and after treatment

The hard materials chosen have shown very good improvement in hardness irrespective of the material type due to adaption of cryogenic treatment. This improvement is in respect of transformation of austenite into martensite. The increase in hardness is due to the decrease in the level of retained austenite. The austenite level is lead by the addition of nickel, chromium or molybdenum. This statement is supported by X-ray data of retained austenite.

3.5. Microstructure

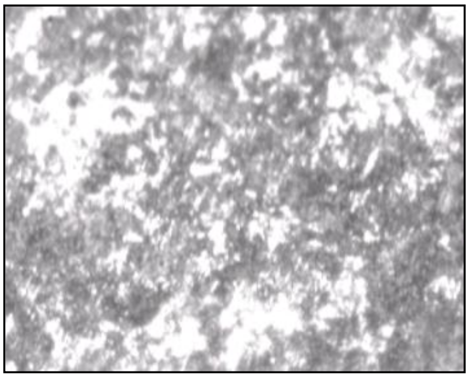

Fig 4: Microstructure of spring steel before treatment

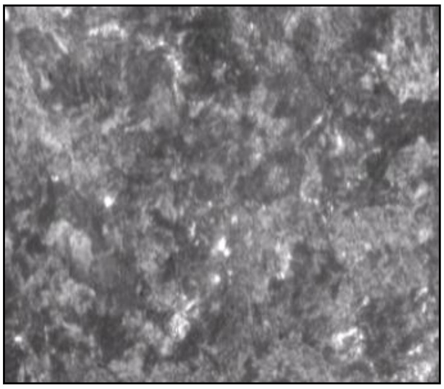

Fig 5: Microstructure of Spring steel before treatment

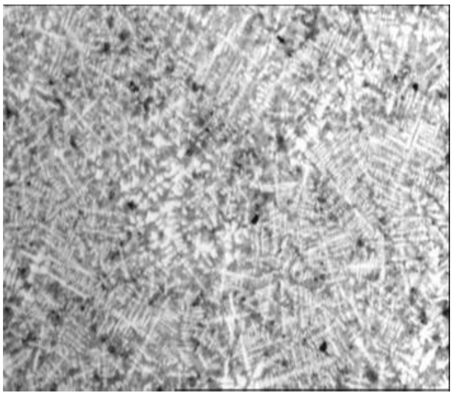

Fig 6: Microstructure of High Chromium steel before treatment

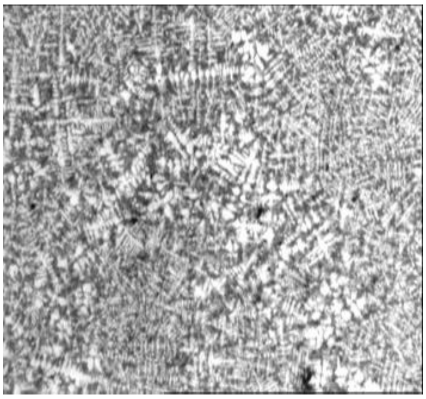

Fig 7: Microstructure of High Chromium steel after treatment

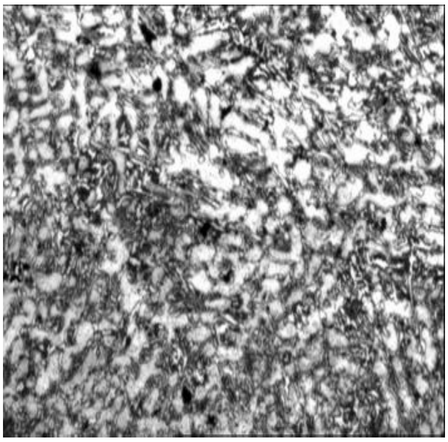

Fig 8: Microstructure of Nihard4 steel before treatment

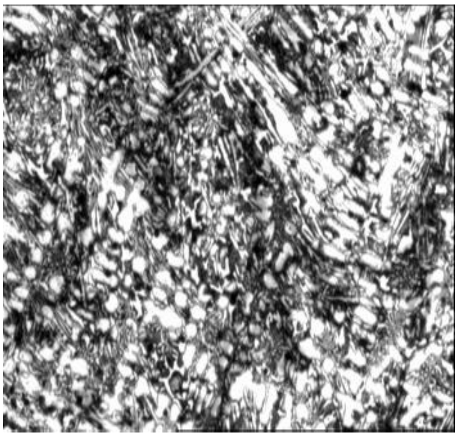

Fig 9: Microstructure of Nihard4 steel after treatment

The micro-structure of the samples before cryogenic treatment and the samples after cryogenic treatment were examined under the optical microscope which shows predominating martensic structure with some amount of retained austenite. As seen in the above figures the white portions represent the austenite rich regions in all the specimens before the cryogenic treatment which gets converted dense martensic structure after the cryogenic treatment which is has shown. This phase transformation enhances the desirable mechanical properties of all materials.

4. CONCLUSION

It can be inferred from the investigation that the cryogenic treatment carried out on spring steel is quite effective by way of improving the hardness and wear resistance properties. For application involving slide wear it is beneficial to adapt the cryogenic process for wear life improvement.

- Hardness is higher for cryogenically treated sample than the untreated samples.
- There is a reduction in the retained austenite content in the cryogenically treated samples.
- Optical features reveal fine martensic structure with very low retained austenite content.
- Residual stresses have reduced in the cryogenically treated samples when compared to untreated samples.
- Cryogenic treatment has thus improved the overall material characteristics.

REFERENCES

[1]A.Y.L. Yong et al, “Performance evaluation of cryogenically treated tungsten carbide tools in turning” International journal of Machine tools and manufactures 46 2051-2056 (2006)

[2] K.H. W. Seah et al, “Performance evaluation of cryogenically treated tungsten carbide cutting tool inserts” Journal of Engineering Manufacture Vol 217 (2003)

[3] Nadig D.S, Jacob S. Karunanithi R, Manjunatha R, Subramanian D, Prasad M.V.N, Geetha Sen, Abhay K.Jha. “Studies of Cryotreatment on the Performance of Integral Diaphragm Pressure Transducers for Space Application”.

[4] A. Bensely, Pete Paulin, G.Nagarajan and D. Mohan Lal “Dimentional Stability in cryogenic Treatment”.

[5] Bensely A., Venkatesh S., Mohan Lal D., Nagarajan G., Rajadurai A. and Krzysztof Junik, ‘Effect of cryogenic treatment on distribution of residual stress in case carburized En 353 steel’, Materials Science and Engineering: A, Volume 479, Issues 1-2, 25 April 2008, pp. 229-235.

[6] W. Wu, L.Y. Hwu, D.Y. Lin and J.L. Lee, The Relationship Between Alloying Elements And Retained Austenite In Martensitic Stainless Steel Welds, October 5,1999, 42 (2000) 1071–10