Impact Behavior of Cryogenically Treated En 52 and 21-4N Valve Steels

M. Arockia Jaswin, D. Mohan Lal

Abstract**—**Cryogenic treatment is the process of cooling a material to extremely low temperatures to generate enhanced mechanical and physical properties. The purpose of this study is to examine the effect of cryogenic treatment on the impact behavior of En 52 and 21-4N valve steels. The valve steels are subjected to shallow (193 K) and deep cryogenic treatment (85 K), and the impact behavior is compared with the valve steel materials subjected to conventional heat treatment. The impact test is carried out in accordance with the ASTM E 23-02a standard. The results show an improvement of 23 % in the impact energy for the En 52 deep cryo-treated samples when compared to that of the conventionally heat treated samples. It is revealed that during cryogenic treatment fine platelets of martensite are formed from the retained austenite, and these platelets promote the precipitation of fine carbides by a diffusion mechanism during tempering.

Keywords**—**Cryogenic treatment, valve steel, Fractograph, carbides, impact strength.

I. Introduction

The service failures of engineering components have always been a challenge to metallurgists. Apart from various production methods, using various processes for improving the life of components can reduce this failure. Cryo-processing can produce harder, more wear-resistant materials with many other beneficial properties [1]-[3]. Cryogenic treatment is a process attempted by researchers to supplement the conventional heat treatment to improve the mechanical properties of materials. Cryogenic treatment, unlike coatings, is an inexpensive one-time permanent treatment affecting the entire section of the component. The treatment is an add-on process over the conventional heat treatment in which the samples are cooled down to the prescribed cryogenic temperature level at a slow rate, maintained at this temperature for a long time and then heated back to room temperature, and tempered to decrease the brittleness of the martensite [4]. The notable effects of the cryogenic treatment include changes in the mechanical properties and the microstructure of materials. The refrigeration of metals to improve their performance is generally classified as either shallow cryogenic treatment (SCT), sometimes referred to as subzero treatment, or deep cryogenic treatment (DCT) based on the treatment temperature [5]. Common practice identifies 193 K (- 80^{o}C) as the optimum temperature for SCT at which the parts are held (soaked) for 1 hour per inch of thickness, and then subsequently warmed in ambient air. Deep cryogenic treatment in the range of -125 to -196^{o}C improves certain properties beyond the improvement obtained by shallow cryogenic treatment. The theories concerning the effects of cryogenic treatment are the more nearly complete transformation of retained austenite into martensite, and the strengthening of the material brought about by the precipitation of submicroscopic carbides [6], [7]. Depending on the alloy composition of the metals and the pre-hardening cycles, the benefits reaped are increased strength, greater dimensional stability or microstructural stability, improved wear resistance and relief of residual stress [8]. The effect of shallow cryogenic treatment (SCT) at 193 K and deep cryogenic treatment (DCT) at 77K on the microstructure and toughness of the En 31 bearing steel was studied by Harish et al. [9]. It was reported that the toughness of the bearing steel remains almost the same after both deep and shallow cryogenic treatment. The microstructural study of the En 31 bearing steel concluded that the cryogenic treatment should be followed by tempering to promote secondary carbide precipitation, which is essential for hardness augmentation and wear resistance improvement. The static mechanical properties of a commercial gear carburized steel (18NiCrMo5) were investigated by Paolo Baldissera and Cristiana Delprete [2] through hardness and tensile tests followed by optical fractographic observations. The results pointed out a substantial hardness improvement for all the cryotreated groups and a remarkable enhancement of the tensile strength. Johan Singh et al. [10] investigated the fatigue life extension of notches in AISI 304L weldments using deep cryogenic treatment. The fatigue crack initiation life from notches of cryogenically treated samples was found to have improved. The strain-induced martensite formed by cryogenic treatment is considered to be effective on the fatigue initiation life extension in the high cycle regime. The fatigue crack propagation properties were not improved due to this cryogenic treatment.

Research efforts are widespread with a view to improving the life and performance of components on automotive applications by various treatments. Recent work has shed light on the effects of cryogenic treatment on bearings, gears and engine components to reduce wear, and improve performance [1], [6], [9], and [11]. The requirements for automotive engines in recent years have included higher horsepower, lower fuel consumption, and longer maintenance-free life. These requirements have increased the severity of the engine

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valve and valve seat insert wear. The valve and valve seat are the key parts installed in the cylinder head of an engine to ensure the sealing of the engine chamber. Xia Yong et al. [12] reported that many faults of engines occur on the valve train. Although new valve materials and production techniques are constantly being developed, these advances have been outpaced by demands for increased engine performance, and wear related problems, and failure of engine valves remain an issue. Since research efforts are widespread with a view to improving the life and performance of components in automotive applications by various treatments, this cryogenic treatment will give an appropriate solution. A study conducted previously by the same authors on the effect of cryogenic treatment on the wear resistance of En 52 and 21-4N valve steels showed a considerable enhancement in the wear resistance through the cryogenic treatment. The benefits attained through cryogenic treatment shown by the above literature made us to execute a study on the critical properties of the valve steels. The effect of cryogenic treatment on impact behavior of valve steel materials has not been investigated so the present study was conducted. The focal point of this experimental study is to investigate the influence of cryogenic treatment on the impact behavior of En 52 and 21-4N valve steels. The metallurgical reason for the increase in the impact properties with regard to each treatment is identified by conducting a fractography study. The En 52 valve steel has been widely used for intake valves and exhaust valve stems in internal combustion engines. The 21-4N valve steel is heat-resistant steel, used as the plate material of diesel engine exhaust valves in the automobile industry. This research finding will definitely be helpful to valve and engine manufacturers to improve the valve quality and life.

II. Experimental Procedure

The chemical compositions of the selected valve steel materials are confirmed using the optical emission spectroscope (OES).

The results obtained by the chemical analysis of the raw materials are listed in Table I. The result of the chemical analysis confirms the chemical compositions of the En 52 and 21-4N valve steels. The specimens required for the impact test are machined as per the ASTM E 23-02a standard [13]. The machined specimens are grouped into three and subjected to three different treatment processes. The conventional heat treatment (CHT) followed for the En 52 and 21-4N valve steels are given to one group of the specimens. The conventional heat treatment for the En 52 and 21-4N valve steels is conducted based on the valve alloy heat treatment procedures discussed by Wang [14] and Nayar [15]. The following heat treatment is given to one group of the En 52 specimens. Hardening (austenitizing) at 1248K (975ºC) for one hour, is followed by oil quench and tempering at 923K (650°C) temperature for one hour. One group of the 21-4N specimens is solution-heated for 40min at 1413K (1140ºC) and then water-quenched; the subsequent ageing is carried out at 1018K (745°C) for 5 hours. The heat treatment and cryogenic treatment for the En 52 and 21-4N valve steels are shown in Fig. 1.

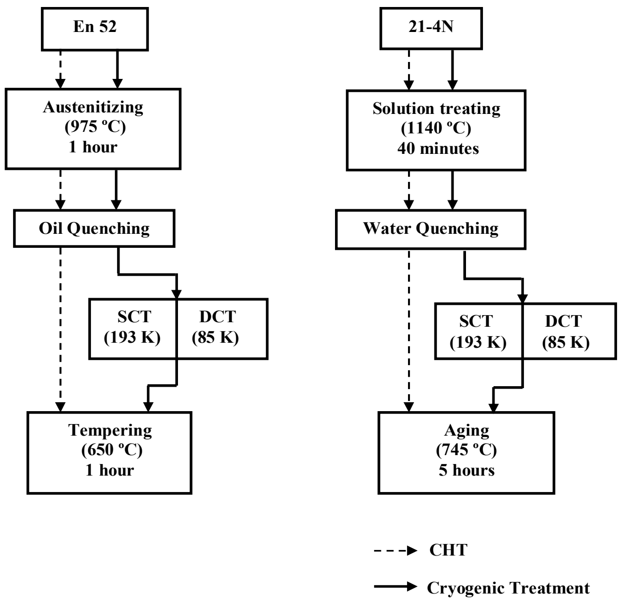

Fig. 1 Treatment processes for the En 52 and 21-4N valve steels

TABLE I
Chemical Composition Of En 52 And 21-4N Valve Steel

Material	Carbon (%)	Silicon (%)	Manganese (%)	Chromium (%)	Nickel (%)	Nitrogen (%)	Sulphur (%)	Phosphorus (%)
En 52	0.45	3.25	0.50	8.50	0.40	-	0.03	0.04
Typical composition	0.4-0.5	2.7-3.3	0.6 max	8-10	0.5 max	-	0.03 max	0.04 max
21-4N	0.48	0.25	8.00	20.00	4.20	0.45	0.03	0.05
Typical composition	0.48-0.58	0.25 max	8-10	20-22	3.25-4.5	0.35-0.5	0.03 max	0.05 max

A. Cryogenic Treatment

Cryogenic treatment is the process of cooling a material to extremely low temperatures to generate enhanced mechanical and physical properties. Two types of cryogenic treatment are given to the valve steel samples, namely, shallow cryogenic treatment (193 K) and deep cryogenic treatment (85 K). The En 52 valve steel samples of one group, which had already undergone quench hardening, are subjected to shallow cryogenic treatment.

In SCT the samples are directly placed in the mechanical freezer at 193K (- 80^{o}C), and soaked for an hour. The samples are removed from the freezer and allowed to reach ambient temperature. They are then tempered at 923K (650°C) for an hour. For the 21-4N valve steel the solutionized samples are kept in the mechanical freezer at 193K (- 80°C), soaked for an hour, and then removed and aged at 1018 K (745°C) for 5 hours.

In deep cryogenic treatment, one group of hardened En 52 & solutionized 21-4N samples are slowly cooled from room temperature to 85K at 1K/minute, soaked at 85K for 24 hours and finally heated back to room temperature at 0.6K/minute. This controlled process is achieved using the computerized control A.C.I. CP-200vi (Massachusetts, U.S.A) cryogenic treatment processor and the photographic image are shown in Fig. 2.

Fig. 2 Cryogenic treatment processor

The processor is a well-insulated chamber with liquid nitrogen as the working fluid. The cryogenic processor consists of a treatment chamber, which is connected to a liquid nitrogen tank (MVE DURA-CYL 160MP) through a vacuum insulated hose. The temperature sensors inside the chamber sense the temperature and accordingly the PID temperature controller operates the solenoid valve to regulate the liquid nitrogen flow. The liquid nitrogen passes through the spiral heat exchanger and enters the duct leading to the bottom of the chamber as nitrogen gas. The blower at the top of the chamber sucks the gas coming out at the bottom and makes it circulate effectively inside the chamber and reduces the chamber temperature. The programmable temperature controller (Partlow 1462) of the cryogenic processor is used to set the deep cryogenic treatment parameters, which in turn, control the process parameters like soaking time, temperature and cooling rate. Through the data acquisition system, the deep cryogenic treatment processes are recorded and stored. The En 52 samples taken out from the processor are tempered at 923K (650°C) for an hour and the 21-4N are aged at 1018 K (745°C) for 5 hours.

B. Impact Test

The Charpy and Izod impact tests are carried out in accordance with the ASTM E 23-02a standard [13]. The En 52 valve steel samples are subjected to the Charpy impact test. The En 52 valve steel is obtained in the form of a 16mm diameter rod, which is milled to the dimensions shown in Fig. 3.

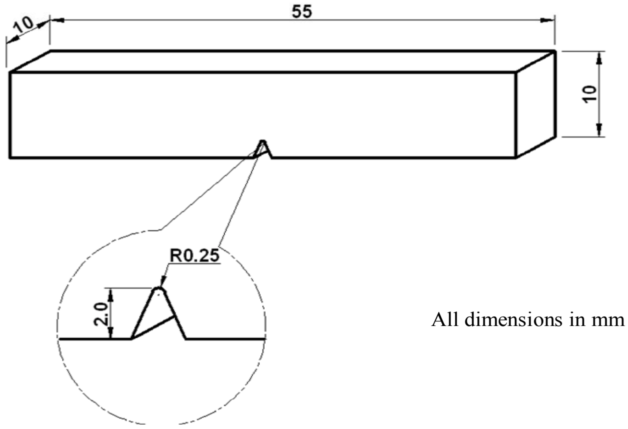

Fig. 3 Impact test specimens for the En 52 valve steel

Twelve samples of the En 52 valve steel are milled to a length of 55mm and a cross section of 10 x10mm, having a V-notch with an angle of 45º and a depth of 2mm. The material available for the 21-4N valve steel is in the form of a 12mm diameter rod, so sub-size specimens as per the standard are milled for conducting the impact test on a low capacity Izod impact testing machine. Fifteen samples of the 21-4N valve steel are milled to the length of 55mm and a cross section of 5 x 5mm having a V-notch with an angle of 45º and depth of 1mm. The machined samples are grouped into three with five in each group, and subjected to CHT, SCT and DCT respectively. The Charpy and Izod impact test samples of each group are subjected to the impact test as per the ASTM E23-02a [13]. The test is carried out at an ambient condition. This helps to identify the best treatment for En 52 and 21-4N valve steels with respect to the impact behavior. The study also helps in identifying the metallurgical reason for the increase or decrease in the impact properties with regard to each treatment.

C. Fractography Study

The scanning electron microscopic analysis helps to characterize the fracture surfaces of the broken specimen after the impact tests at extremely high magnifications, and also to view the unseen surface features. The Scanning electron microscope (SEM) operating at 20 kV is used to observe the morphology of the fractured specimens. In the present investigation, the SEM analysis is carried out to characterize the fracture surfaces of the broken specimen after the impact tests. The fractured surfaces are viewed at 1000X magnification. This helps in identifying the mechanism involved. The examinations are carried out using a HITACHI scanning electron microscope (5 X to 300000 X).

III. Results and Discussion

The results of the Charpy impact test for the En 52 and Izod impact test for the 21-4N valve steel conducted on the CHT, SCT and DCT specimens are given in Tables II and III. The results show an increase in toughness after SCT and DCT when compared with that after CHT. The average impact energy for the CHT sample is 2.9 J; for the SCT sample it increased slightly to 3.1 J, and for the DCT sample it further increased to 3.57 J. The improvement in the impact energy for the DCT samples is 23.1% when compared to that of the CHT samples.

TABLE II
Charpy Impact Test Results for the EN 52 Valve Steel Subjected to the CHT, SCT and DCT

S.No.	IMPACT ENERGY (J)		
	CHT	SCT	DCT
1	2.8	3	3.7
2	2.9	3.2	3.7
3	3	3.2	3.4
4	2.9	3	3.5
Average	2.9	3.1	3.57

The average impact energy obtained from the Izod impact test of the 21-4N material is 1.72 J for the CHT samples, 2.06 J for the SCT samples and 2.2 J for the DCT samples. When comparing the absorbed energy, the DCT samples absorb more energy than the SCT and CHT samples. The possible reason for the improvement in toughness may be due to the relieving of residual stress as a result of lattice level contraction and expansion during the cool down and warm up processes of the cryogenic treatment, or due to the precipitation of fine carbides during the subsequent tempering/aging.

TABLE III
Izod Impact Test Results for the 21-4N Valve Steel Subjected to the CHT, SCT and DCT

S.No.	IMPACT ENERGY (J)		
	CHT	SCT	DCT
1	1.9	2.2	2.1
2	1.5	2	2.3
3	1.5	2.2	2.1
4	1.8	2	2.2
5	1.9	1.9	2.3
Average	1.72	2.06	2.2

The cryogenic treatment increases the driving force for the nucleation of carbides, and facilitates the precipitation of a higher number of finer carbides, resulting in higher toughness values. During cryogenic treatment fine platelets of martensite are formed from the retained austenite, and these platelets promote the precipitation of fine carbides by a diffusion mechanism during tempering; it may also be due to the reduction in lattice energy during cryogenic treatment, which makes the crystal structure perfect. The fractograph of the En 52 CHT, SCT and DCT specimens taken at the central region of the Charpy impact test are shown in Fig. 4, 5 and 6 respectively. The fracture surface of the CHT specimen shows a transgranular brittle fracture with a small amount of plastic deformation. Depressions and plateaus on the fracture surface reveal a reduction in the plastic deformation of the CHT specimen. Figs. 5 and 6 show the regions with both intergranular fracture and dimple, which quantify the plastic deformation of the specimen.

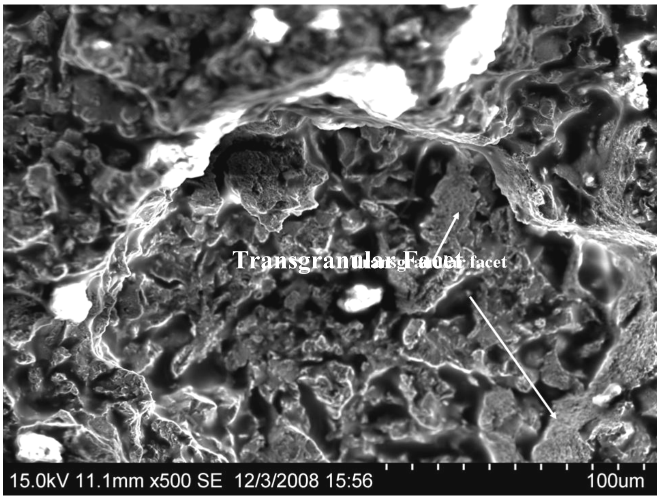

Fig. 4 Fractured image of the En 52 CHT specimen

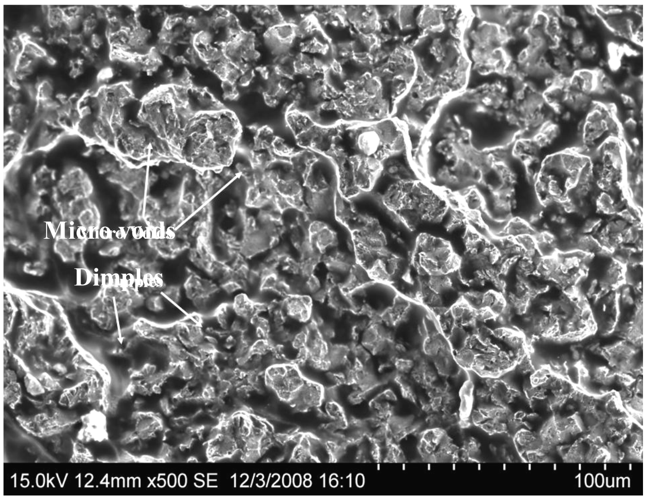

Fig. 5 Fractured image of the En 52 SCT specimen

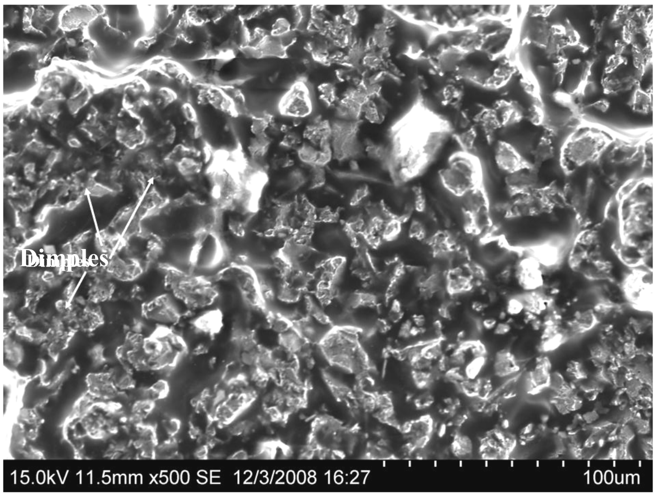

Fig. 6 Fractured image of the En 52 DCT specimen

The fractograph shown in Fig. 6 indicates the decreased size of the acicular needles of martensite as a result of the DCT; this enhances the toughness of the matrix. Figs. 7-9 show the fracture surface of the 21-4N CHT, SCT and DCT specimens respectively. The facets present in the fracture surface of the CHT specimen are more, compared to those of the SCT and DCT specimens. In the fractured surface of the DCT specimen, more secondary cracking is observed, and it is due to severe bubble coalescence along the grain boundaries. The presence of microdimples on the fracture facets shows that a considerable amount of plastic deformation has occurred prior to the fracture resulting in increased impact energy of the cryo-treated specimen. The presence of many microcracks and crack branching indicates an increase in the impact energy over that of the conventionally heat treated samples.

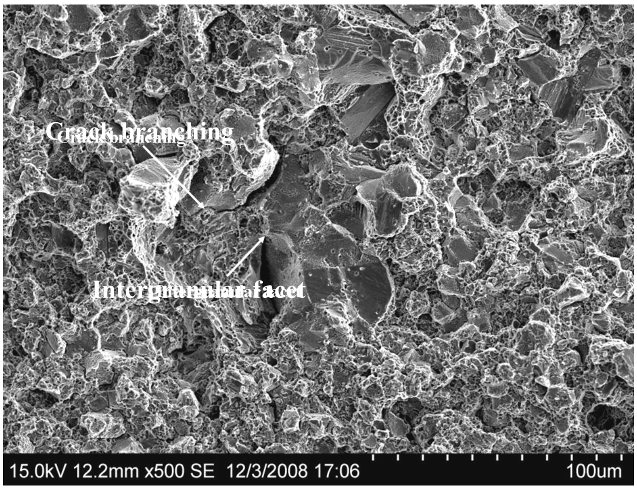

Fig. 7 Fractured image of the 21-4N CHT specimen

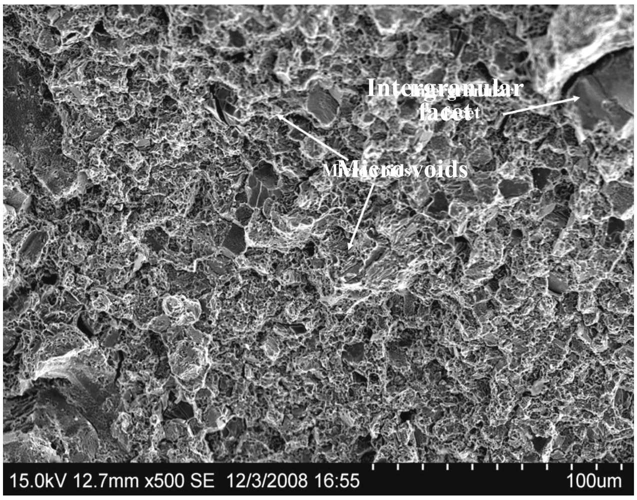

Fig. 8 Fractured image of the 21-4N SCT specimen

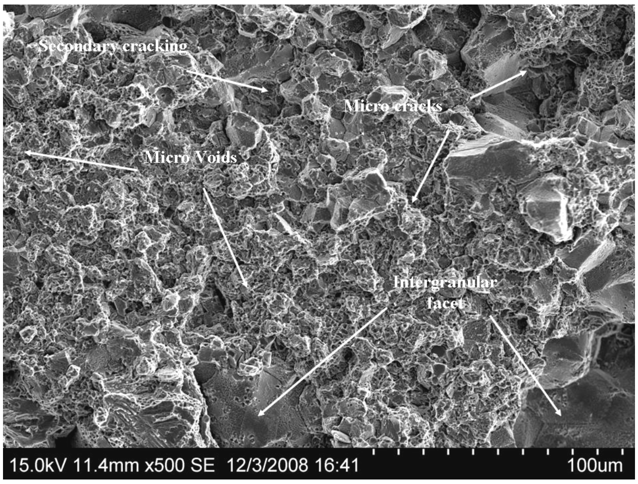

Fig. 9 Fractured image of the 21-4N DCT specimen

IV. Conclusion

It is concluded that deep cryogenic treatment has the potential to improve the impact behavior of valve steels.

- ❖ The improvement in the impact energy of the En 52 DCT samples is 23 % when compared to that of the CHT samples. Cryogenic treatment increases the driving force for the nucleation of carbides, and facilitates the precipitation of a higher number of finer carbides, resulting in higher toughness values.
- ❖ Fine platelets of martensite are formed from the retained austenite, and promote the precipitation of fine carbides by a diffusion mechanism during tempering.

The fractured surface of the DCT specimens show more secondary cracking, and it is due to severe bubble coalescence along the grain boundaries. The presence of microdimples on the fracture facets shows that a considerable amount of plastic deformation has occurred prior to the fracture resulting in increased impact energy of the cryo-treated specimens. This study confirms that deep cryogenic treatment can very well be adopted for improving the impact strength of these valve steels.

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