Influence of cryogenic treatment on the dimensional stability of Gear Steel-En 353

[1]Bensely A., [2]Pete Paulin, [3]Nagarajan G., [3]Mohan Lal D
[1]3012 Shadowood Pkwy SE, Atlanta, Georgia 30339, USA.
[2]300 Below, Inc., 2999 E. Parkway Dr., Decatur, Illinois, 62526,USA.
[3]Department of Mechanical Engineering, Anna University, Chennai, India.

Case carburized steel (En 353) when cryogenically treated showed tremendous improvement in wear resistance [1]. This is due to the microstructural changes such as conversion of retained austenite to martensite and precipitation of fine alloying carbides. The equivalent grades of En 353 are BS-815M17 and IS-15NiCr1Mo12. In an earlier research work on En 353 it was found that cryogenic treatment changes the residual stress [2]. This research article describes the dimensional stability of En 353 steel after Conventional Heat Treatment (CHT), Shallow Cryogenic Treatment (SCT) and Deep Cryogenic Treatment (DCT). Coefficient of linear thermal expansion is very much used for design purpose and to determine failure by thermal stresses when a material is subjected to temperature variations. Thermomechanical analysis was carried out as per ASTM E 831 for studying the linear thermal expansion of En 353 [3].

Heat Treatment of En 353

Heat treatment is a combination of heating and cooling applied to a metal or alloy in the solid state in a way that will produce desired effects on the properties. All basic heat-treating processes for steel involve the transformation or decomposition of austenite. The first step in the heat treatment of steel is to heat the raw material to a temperature nearer or above the critical temperature in order to form austenite. The nature and appearance of this transformation determines the physical and mechanical properties of any given steel. Gears are generally subjected to carburization process to achieve the desired properties. The basic principle of carburizing has remained unchanged, since carburizing was first employed. The method used to introduce the carbon into the steel has been a matter of continuous evolution. In its earliest application, components were simply placed in a suitable container and covered with a thick layer of carbon powder (pack carburizing). Although effective in introducing carbon, this method was exceedingly slow, and as the demand for production grew, a new process using liquid and gaseous atmosphere was developed. They are gas carburizing, vacuum carburizing, plasma carburizing and salt bath carburizing.

Conventional Heat Treatment (CHT) Process

Numerous industrial components such as gears, crown wheel and pinion require a hard wear resistant surface and a soft tough core to withstand heavy loading. This can be achieved through case hardening process. When En 353, a low carbon steel is subjected to carburizing, a hard wear resistant surface called the case having high carbon content and a relatively soft-tough inside called the core having low carbon content is produced. The two regions (case and core) have different in-service functions to perform.

The conventional heat treatment cycle of En 353 begins with gas carburizing and is followed by air cooling, quench hardening in oil and tempering. In the present study liquid carburization process was employed instead of gas carburization to achieve a

case depth of 1 mm. Liquid carburized cases are higher in carbon and lower in nitrogen. It has the advantages of freedom from oxidation, sooting problems, production of uniform case depth and carbon content, a rapid rate of penetration and reduction of time required for the steel to reach carburizing temperature. The carburizing environment was created by fusing a mixture of sodium cyanide, potassium chloride, sodium chloride and sodium cyanate. The machined raw specimens(5 mm diameter and 5 mm height) were placed in a bath of molten cyanide, so that carbon will diffuse from the bath into the metal and produce a hard case. Cyanide oxidizes at the surface of the steel producing carbon monoxide, which in turn dissociates to carbon dioxide and nascent carbon with rapid diffusion into steel at high temperatures. In conventional heat treatment process the carburizing temperature was 1183 K and the cycle time for carburizing was 5 hours. At this temperature the following reaction takes place.

$$Fe + 2CO \rightarrow Fe\,(\,C\,) + CO_2$$

where Fe(C) represents carbon dissolved in austenite. Due to this carburization, a surface layer of high carbon equivalent to the carbon potential (0.75 %) is quickly built up in the case. Since the raw material has low carbon content (0.17%), the carbon atoms try to reach equilibrium by diffusing inward. After diffusion has taken place for the required amount of time, depending upon the case depth desired (1 mm), the component was removed from the furnace. The material after carburizing was cooled in air. It was followed by quench-hardening process. In quench-hardening process, the carburized specimens were heated to 1093 K (1508^0F) and soaked for 30 minutes and suddenly quenched in oil at 313 K(104^0F). Gears are generally oil quenched to avoid distortion to the lowest possible level. This rapid cooling from the hardening temperature causes the transformation of austenite, which is soft and ductile, into martensite, which is very hard and brittle. This also suppresses the conversion of austenite into ferrite and cementite. Hence the structure of hardened steel consists of mainly tetragonal martensite and some amount of retained austenite that depends on the chemical composition of steel. This transformation is diffusionless and time independent and there is no change in chemical composition.

The hardening of steel depends entirely on the formation of martensite. It increases the compressive strength and wear resistance of steels, but by itself leaves the steel very brittle. Tetragonal martensite and retained austenite are unstable. Therefore hardened steel naturally has a tendency to pass into equilibrium or stable condition. But this equilibrium is not reached at room temperature because of low mobility of atoms at this temperature. So, after quench-hardening, the samples were immediately subjected to tempering. As the temperature rises during tempering the mobility of atoms increases causing phase and structural changes until reaching equilibrium. For the present investigation the quench hardened samples are heated to 423 K (302^0F) and soaked for 1.5 hours. During this process the carbon atoms separates from the space lattice of tetragonal martensite. Consequently the martensite breaks down into a transition precipitate known as iron carbide (Fe_3C) and a low carbon martensite (soft structure). During this the internal stresses get removed and the ductility increased. Tempering was carried out to gain toughness and relive internal stresses at the cost of some hardness and tensile strength.

Cryogenic Treatment Process

The retained austenite present in the conventional heat treatment process after hardening can be alleviated by means of cryogenic treatment. Since in most of the alloy steel martensite finish temperature does not lie above room temperature the problem of retained austenite in service still prevails. Hence the steel has to be cooled still further down from room temperature to achieve 100% martensite. Cryogenic treatment is an extension of conventional heat treatment to achieve 100% martensite. This treatment alters the material microstructures that enhances strength and wear resistance. For maximum benefits cryogenic treatment should be introduced between hardening and tempering process. Hardened alloy steels such as carburized gears, pinion and shafts are particularly responsive to this treatment. Depending on the alloy composition 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 [1, 2, 4, 5].

Presently two major types of cryogenic treatment are available in practice. They are SCT and DCT. There is a lot of confusion among researchers in classifying the temperature applied in both the treatments. Hence it becomes imperative to define both in order to enable readers to identify the temperature limits referred in this paper. In order to have clarity the details of conventional heat treatment and cryogenic treatment parameters adopted for En 353 in the present study is shown in Figure 1.

Shallow Cryogenic Treatment (SCT)

Due to high carbon content attained in the case during carburization there is retention of austenite in En 353, which can transform during service. This is detrimental to the material. Hence in the present work specimens which have undergone conventional hardening process (i.e., oil quenching) were directly kept in freezer at 193 K(-112^0F) for 5 hours to complete the martensitic transformation. It was followed by tempering at 423 K(302^0F) for 1.5 hours . It was done to ensure that there is no brittle untempered martensite when the component is put into service.

Deep Cryogenic Treatment (DCT)

Similar to shallow cryogenic treatment, deep cryogenic treatment is also a supplementary process to conventional heat treatment. Even though the martensite finish temperature (M_f) lies nearer to 193 K(-112^0F), the need for deep cryogenic treatment is due to the increased benefits reported by Barron (1974) on other materials when treated at 77 K(-321^0F) [5]. DCT is expected to enhance the desired metallurgical and structural properties by completing the transformation of austenite (a softer metal phase) to martensite (a tougher, more durable metal phase). In the present investigation the material which has undergone conventional hardening was cooled from room temperature to 77 K (-321^0F) in 3 hours, soaked for 24 hours and heated back to room temperature in 6 hours. These very low temperatures were achieved using computer controls in a well-insulated treatment chamber with liquid nitrogen as working fluid. Liquid nitrogen, being cheap, inert and easily available is highly suitable to attain 77 K (-321^0F). After the specimens reach room temperature it was immediately subjected to tempering at 423 K (302^0F) for 1.5 hours in order to attain carbide precipitation and tempered martensite.
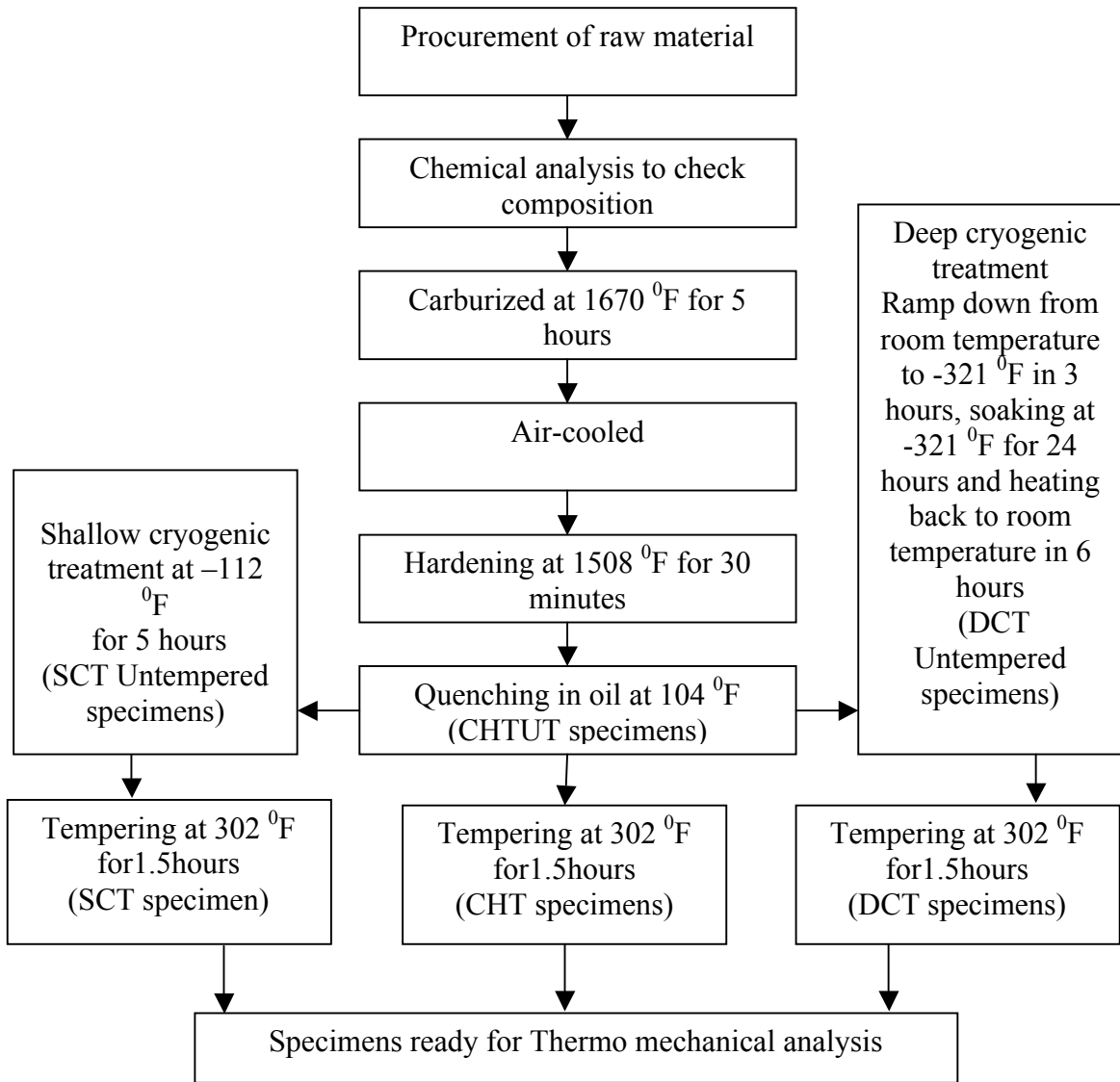
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                    ┌─────────────────────────────┐
                    │  Procurement of raw material │
                    └─────────────────────────────┘
                                   │
                                   ▼
                    ┌─────────────────────────────┐
                    │  Chemical analysis to check  │
                    │         composition          │
                    └─────────────────────────────┘
                                   │
                                   ▼
                    ┌─────────────────────────────┐
                    │  Carburized at 1670 $^0$F for 5 │
                    │            hours             │
                    └─────────────────────────────┘
                                   │
                                   ▼
                    ┌─────────────────────────────┐
                    │          Air-cooled          │
                    └─────────────────────────────┘
                                   │
                                   ▼
                    ┌─────────────────────────────┐
                    │ Hardening at 1508 $^0$F for 30 │
                    │           minutes            │
                    └─────────────────────────────┘
                                   │
                                   ▼
                    ┌─────────────────────────────┐
                    │  Quenching in oil at 104 $^0$F │
                    │       (CHTUT specimens)      │
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Deep cryogenic treatment Ramp down from room temperature to -321 ^{0}F in 3 hours, soaking at -321 ^{0}F for 24 hours and heating back to room temperature in 6 hours (DCT Untempered specimens)

Shallow cryogenic treatment at −112 ^{0}F for 5 hours (SCT Untempered specimens)

Tempering at 302 ^{0}F for 1.5 hours (SCT specimen)

Tempering at 302 ^{0}F for 1.5 hours (CHT specimens)

Tempering at 302 ^{0}F for 1.5 hours (DCT specimens)

Specimens ready for Thermo mechanical analysis

Figure 1   Heat treatment and cryogenic treatment steps adopted for the study

Results and Discussion

Materials expand to some extent when heated. The heat increases the average amplitude of vibration of the atoms, which in turn increase the average separation. Suppose an object of length L undergoes a temperature change of magnitude ΔT and if ΔT is reasonably small, the change in length, ΔL, is proportional to L and ΔT. Hence

$$\alpha = \frac{\Delta L}{L \Delta T}$$

where α is called the coefficient of linear expansion for the material. The specimen to be tested was machined to 5 mm in length and 5 mm in diameter and subjected to CHT, SCT and DCT processes. The dimensional stability of En 353 after CHT, SCT and DCT were studied using a Mettler thermo mechanical analyzer. The treated specimen was placed in the specimen holder under the sensing probe, with the

temperature sensor in contact with the specimen. The furnace encloses the specimen holder. An appropriate load force of 20mN wan applied to the sensing probe to ensure that it was in contact with the specimen. The specimen was heated at a constant heating rate of 20 K/minute from room temperature to 1103 K (1526°F). The change in the specimen length was recorded using linear variable differential transformer and the data obtained helps in comparing and describing the dimensional stability of En 353 during service. The linear expansion coefficient estimated for the CHT, SCT and DCT samples are shown in Figure 2.
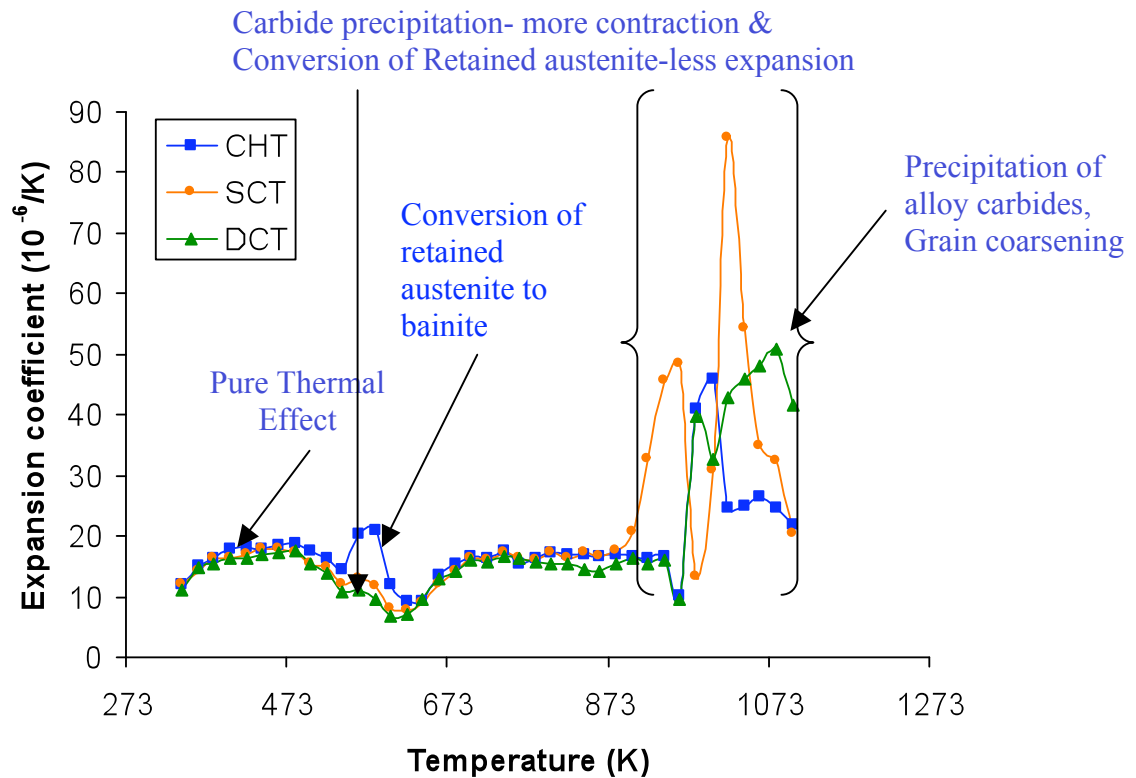

Figure 2   Linear expansion coefficient for CHT, SCT and DCT specimens

The coefficient of thermal expansion (CTE) is low for the DCT specimens, which can indicate that the dimensional stability is high. The CTE increases in the three specimens till 503 K (446°F) and remains the same, which is due to pure thermal effect. A redistribution of the carbon atoms in the martensite occurs roughly from room temperature to 373 K (212°F). This can be due to both segregation and movement of carbon atoms to the lattice defects such as dislocations and twin boundaries or by the clustering of carbon atoms, which in turn can occur in several ways and involve spinodal decomposition and ordering. From temperature 503 K (446°F) to 643 K (698°F), a decrease in CTE is observed in SCT and DCT specimens. During this period an increase and decrease in dimension in CHT sample takes place. This is due to the conversion of retained austenite to bainite in CHT specimens. X-Ray diffraction studies show that a small amount of retained austenite still prevails in SCT and DCT specimens. Between 503 K (446°F) to 643 K (698°F), SCT and DCT specimens undergo contraction. This is due to the simultaneous conversion of retained austenite to martensite (expansion) and decomposition of martensite into cementite (contraction). The contraction exceeds the expected expansion due to the conversion of small amount of retained austenite left in the specimen, which is a clear indication of large carbide precipitation in SCT and DCT specimens. From 643 K (698^{0}F) to 883 K (1130^{0}F) the CTE remains the same for

the entire specimen. Beyond 883 K (1130^0F) the CHT, SCT and DCT specimens undergo irregular expansion and contraction and can be attributed to the precipitation of metal carbides and grain coarsening.

Conclusions

This study clearly shows that both shallow cryogenic treatment and deep cryogenic treatment aid further conversion of retained austenite to martensite which on tempering will lead to enhanced carbide precipitation. The study also concludes that the dimensional stability of SCT and DCT specimens are higher than CHT specimens. Therefore case carburized gear which demands high wear resistance and good dimensional stability should be subjected to deep cryogenic treatment.

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