Residual Stresses and Dimensional Changes Related to the Lattice Parameter Changes of Heat-Treated JIS SKD 11 Tool Steels

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The residual stresses and dimensional changes related to the lattice parameter of the JIS SKD11 tool steel have been investigated for both conventional and cryogenic treatments. As trapped carbon atoms are released from the martensite, the martensite c/a ratios and dimensions of the specimens treated conventionally decreased at tempering temperatures above 673 K. However, these values increased owing to the increase in the c-axis when tempering was conducted at temperatures higher than 673 K. At higher temperatures, the retained austenite is converted into martensite. The martensite c/a ratios and dimension change ratios in the cryogenically treated specimens decreased significantly with the tempering temperature because the amount of retained austenite is reduced by cryogenic treatment. In the conventionally treated specimens, the compressive residual stress decreased with quenching after tempering because of the release of trapped carbon atoms. However, the retained austenite to martensite transformation and the precipitation of alloy carbides leads to increased martensite lattice parameters and dimensional residual stress changes. The residual stress is also changed to generate compressive residual stresses. The dimension change ratio in cryogenically treated and tempered specimens decreased as the tempering temperature increased through a reduction in the amount of trapped carbon. However, the magnitude of the compressive residual stress was reduced to zero.

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1. Introduction

Quenched, cold-worked steel JIS SKD 11 can be tempered at low temperatures (<453 K) to produce a high hardness (>61 HRC) material. However, this steel may contain a significant amount of austenite retained in the martensite matrix. There is considerable interest in the relationship between the retained austenite transformation and the associated dimensional changes for the production of precision mechanical parts, such as punch dies and gauges. Many researchers believe that retained austenite negatively affects the dimensional stability of tool steel.1,2) Cryogenic treatment and high-temperature tempering can transform the retained austenite into martensite and precipitate fine carbides into the martensite matrix.3) The potential for distortion in heat-treated automotive component manufacturing also depends on the material properties induced in previous stages of raw steel manufacturing.4)

The increments by heat treatments give rise to dimension expansion caused by the austenite transforming into martensite. This can result in a 4.2% increase in volume and a 1.4% increase length. Thermal stresses and phase transformation stresses are the two main causes of distortion during heating and cooling. The dimensional variations caused by phase transformation are affected by metal elements other than carbon itself. In particular, the transition element atoms contained in the carbides in SKD 11 steel, such as chromium, molybdenum and vanadium, will be dissolved and enriched in the matrix when the SKD 11 steel is austenitized. This substitutional solid-solution mechanism distorts the austenite lattice. The carbides that precipitate during tempering also influence the carbon concentration in the martensite lattice. High-temperature tempering and cryogenic treatment research has recently suggested that a reduction in the amount of retained austenite is beneficial to dimensional stability. Another factor beneficial for dimensional stability is the relaxation of residual stresses in the processed steel. 823 K is the most widely applied tempering temperature that activates the secondary hardening mechanism. It reduces the original stresses by 70–90%, leading to greater dimensional stability.1,2) Surberg found that the greatest dimensional stability was achieved using a deep cold treatment at cryogenic temperatures, and that the cryogenic temperature treatment process is time dependent.2) Gogte reported that the period for which the samples are held at low temperature, known as the soaking period, is necessary to facilitate the transformation of the retained austenite into martensite and allow fine carbides to precipitate into the crystal lattice to achieve the lowest possible energy state.5) Pellizzari found that fine cementite precipitated from the martensite and retained austenite, and that this decomposition influences the dimensional changes.6) Residual compressive stresses produced during the rolling process impact the steel in the circumferential and axial directions with respect to the worked track. The quenching process applied to SAE 52100 steel rods resulted in different residual stress and retained austenite distributions at the surface and in the sub-surface regions.7)

Here, an attempt is made to quantify the effect of the heat treatment parameters on the residual stresses and dimensional changes related to the martensite lattice parameters for JIS SKD 11 tool steel. Both conventional and cryogenic treatments were conducted to evaluate the differences between them.

2. Experimental Procedure

The chemical composition of SKD 11 cold-worked steel is listed in Table 1. The SKD 11 steel specimens with dimensions of φ 25 mm × t 50 mm were machined from φ 26 mm as-received steel rods. The specimen surfaces were polished to a surface roughness (Ra) of near-mirror grade, approximately 0.2 Ra.
A two-chamber vacuum furnace incorporating a heating chamber was employed in this experiment. The specimens were preheated at 923 and 1123 K for 25 min each and then austenitized at 1298 K for 20 min. After that, the specimens were removed from the heating chamber and quenched in a cooling chamber containing nitrogen gas at 3 bar. The quenched specimens were double tempered in the range 473 to 923 K; these specimens are designated “conventional”. Some quenched specimens were cryogenically treated, first at a cooling rate of 1 K/min, then held at 113 K for one hour, and then tempered; these specimens are designated “cryogenic”. Tempering must be performed prior to cryogenic treatment to avoid cracking.

The specimens were etched using a 5% Nital reagent for microstructural analysis. The volume fraction of retained austenite was determined by an X-ray diffraction (XRD) analysis conducted with Cu radiation at a 1°/min scanning rate. The retained austenite content in the tool steel was calculated using the XRD technique to subtract the primary carbide amount in the tool steel in order to avoid unexpected errors. The primary carbide content in the tool steel was determined by etching with Beraha’s B0 reagents to clearly identify the carbides.8) The martensitic lattice parameters were obtained by calculating the tetragonal lattice of the (200) and (211) peaks for $c/a$ values.

The bulk hardness values of the samples with different heat treatments were determined using a Rockwell hardness tester after polishing the specimens. A Mitutoyo micrometer with a resolution of 1 µm was used to measure specimen dimensions before and after each treatment. The residual stress measurements were performed using the PULSTEC μ-X360 portable X-ray residual stress analyzer according to the cos$\alpha$ method and using Cr radiation. Diffraction intensity profiles were computed in the radial direction from the measured Debye ring image. All residual stress values were measured at the sample surface in both the transverse and longitudinal directions.

3. Results and Discussion

3.1 Metallography

Figure 1 shows the microstructure of the as-received SKD 11 tool steel. In Fig. 1(a), the bigger primary eutectic carbides are rectangular in a longitudinal view, and the carbide length is two to five times the width. It is apparent that the carbides are aligned along the rolling direction. The bigger primary carbides in the transverse direction [Fig. 1(b)] are about 25 to 30 µm and the small carbides are about 1 to 10 µm. These micrographs show a non-uniform distribution of the bigger carbides and fairly uniform distribution of smaller carbides in the ferrite matrix. The number and size of the primary carbides are related to the forging and rolling processes.

The micrographs in Fig. 2 show representative microstructures produced by these experimental conditions. Figures 2(a) and 2(b) show the structures of the quenched and 473 K tempered steel, respectively. Both exhibit nearly the same structure; there are some massive alloy carbides and small spherical carbide grains in the martensite matrix. The grain boundaries of the primary austenite are easily identified in the quenched specimens. The retained austenite is combined with the martensite in quenched SKD 11 steel, since it could not be effectively transformed by the low temperature (473 K). When the quenched specimens were tempered at 773 and 873 K [Figs. 2(c) and 2(d)] the grain boundaries of the tempered martensite matrix disappeared. Instead, the matrix of quenched martensite and retained austenite transformed into tempered martensite, and the boundaries of the fine carbides precipitated in the matrix facilitated the etching effect. Figures 2(e) and 2(f) show the
microstructures present in quenched specimens that were treated cryogenically at 113 K and then tempered at 473 and 783 K, respectively. The microstructures reveal that the cryogenically treated specimens did not exhibit a noticeably different microstructure from those shown in Figs. 2(b) and 2(d), respectively. The number and size of the carbides in the quenched specimens are smaller than those in the as-received steel; this is related to the austenitizing temperature. Comparing the cryogenically processed materials in Figs. 2(c) and 2(f), the optical micrographs fail to bring out any distinctive differences between the microstructures of conventional and cryogenic treated specimens. This is because of the limited optical microscope magnification. Das also reported the same difficulty in differentiating the microstructures of subzero-temperature-treated steel specimens using optical microscopy.9)

3.2 XRD analysis

The XRD results from the specimens quenched and tempered at different temperatures are shown in Fig. 3. The martensite, retained austenite, and carbide peaks can be identified, though some carbide peaks are partially superimposed onto the austenite or martensite peaks. The retained austenite peaks observed in the specimen tempered at low temperature could not be observed in the specimens tempered at temperatures greater than 773 K.

The XRD data reflect the differences in microstructure between martensite and retained austenite at different tempering temperatures. The microstructures in Fig. 2 reveal that the prior austenite grain boundaries disappeared with tempering conducted at a temperature higher than 773 K, and the austenite XRD peak also disappeared at 773 K. This is in good agreement with the optical micrographs shown in the previous session.

The evidence in Fig. 3 shows that the martensite peak angles gradually shifted to higher values in the specimens tempered at higher temperatures. The broadening of the martensite peaks, i.e., the increase in the full width at half maximum, shown in the figure indicates that at lower tempering temperatures the specimens develop a supersaturated martensite lattice. The austenite and martensite lattice parameters were used to estimate the changes in carbon content that are substantially influenced by tempering.10) Iron carbides are precipitated when tempering is conducted at 473 to 573 K, and secondary carbides (alloy carbides) are precipitated at 673 to 873 K.6)

Das categorized the carbides into primary carbides (with size > 5 µm) and secondary carbides (with size ≤ 5 µm).9) Cryogenic treatment promotes the formation of refined secondary carbides and increases the carbide volume fraction, leading to a more uniform carbide distribution compared to that in conventionally heat-treated D2 steel.9) The primary carbides dominated the matrix in this study, as indicated by optical microscopy. The quantities of primary carbides were determined by quantitative measurements conducted on the microstructures. Since the amount of precipitated carbides was so small, they were excluded from the calculation results.8) The retained austenite calculations were based on the integrated intensities of the martensite (200), martensite (211), austenite (200), and austenite (220) peaks.

Figure 4 shows the effect of the tempering temperature on the volume of retained austenite in the specimens with conventional and cryogenic treatments. The quenched specimens possessed 8.8 vol% retained austenite and 16 vol% carbide. Specimens that were conventionally treated showed that the amount of retained austenite decreased from 8.19 to 2.9 vol% during tempering at 773 K. The retained austenite values of the pretreated cryogenic specimens decreased to 3.9 and 1.9 vol% after they were tempered at 473 and 773 K, respectively. These results verify that retained austenite disappeared with tempering at 773 K or cryogenic treatment, similar to the effect of tempering at 473 K in SKD 11 steel.

The relationship of hardness to the tempering conditions is shown in Fig. 5, which indicates that the secondary-hardening effect was present in both the quenched and cryogenically treated samples. The highest hardness was found in the specimen treated cryogenically following a 473 K tempering.
However, the secondary-hardening effect in the cryogenically treated samples was lower because of elimination of retained austenite by the cryogenic process.\textsuperscript{11} This effect was confirmed by this study. The hardness of the cryogenic samples was higher than that of the quenched samples tempered below 723 K. The conventional specimens exhibited a decrease in the amount of retained austenite and precipitated carbides when tempered at temperatures higher than 748 K, and the secondary-hardening effect was also present. However, the secondary-hardening effect in the cryogenically treated samples was lower because the retained austenite was eliminated.

### 3.3 Lattice parameters

The XRD patterns in Fig. 3 show that the peaks shift with tempering temperature and that the results are well correlated with the lattice parameters shown in Fig. 6. The shift in peak angle grows more intense with increasing tempering temperature. The broadening of the XRD peak profiles is caused primarily by the expansion of crystallite size and the strain. A comparison of the (211) martensite peaks indicates that the trapped carbon atoms in the lattice expand the \( c \)-axis during quenching, and the \( c \)-axis parameter is gradually diminished by tempering.

A previous study revealed the presence of martensite after hardening and secondary carbides along with martensite after tempering.\textsuperscript{6} After high-temperature tempering, the martensite has a low carbon content approaching 0.02 mass\% in the matrix. The XRD peak of the high-temperature tempered specimens is nearly the same as that of the as-received steel.\textsuperscript{9} The influence of tempering on the tetragonal distorted martensite corresponds to the trapped carbon atom variation in martensite. For this reason, the phase amounts and lattice parameter of the martensite are also estimated.

The \( c/a \) value of martensite as a function of the martensite \( a \) parameter was calculated from the martensite (200) peak (for \( a \)) and the martensite (211) peak (for \( c \)). It reveals that the \( a \) and \( c \) axes of martensite vary with the tempering temperature. It is well recognized that tetragonal martensite formation is driven by rapid cooling which transforms the austenite into supersaturated martensite. The tetragonal \( c/a \) ratio is closely related to the degree of alloy enrichment in addition to carbon in the austenite phase. The alloying atoms play the role of substituted elements, and the carbon acts as an interstitial element. The \( c/a \) ratio for the conventional specimens decreased until the tempering temperature rose higher than 573 K. The results listed in Table 2 reveal that iron carbide precipitated when the specimens were tempered in the 473 to 573 K range. The carbon content in the martensite expands the \( c \) parameter and reduces the \( a \) parameter.

Retained austenite reduction and alloy carbide precipitation caused by high-temperature tempering are revealed by the microstructure and XRD results. Figure 7 shows that the martensite \( c/a \) ratio of the conventional specimens increased according to the increase in the \( c \)-axis produced during tempering at temperatures higher than 673 K. At that temperature, the retained austenite is converted into martensite. Steels containing some alloying elements still exhibit the

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**Fig. 5** The variation in hardness with tempering temperature for the conventional and cryogenic specimens.

**Fig. 6** The martensite (211) XRD peak for the different samples: (a) as-received and (b)-(g) quenched and tempered at 473, 573, 673, 773, and 873 K, respectively.

**Table 2** Lattice parameters of the \( c \) and \( a \) axes of the martensite versus tempering temperature.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Conventional</th>
<th>Cryogenic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature/K</td>
<td>( c ) axis</td>
<td>( a ) axis</td>
</tr>
<tr>
<td>As Quenched</td>
<td>0.2899</td>
<td>0.2824</td>
</tr>
<tr>
<td>473</td>
<td>0.2888</td>
<td>0.2855</td>
</tr>
<tr>
<td>573</td>
<td>0.2881</td>
<td>0.2854</td>
</tr>
<tr>
<td>673</td>
<td>0.2883</td>
<td>0.2845</td>
</tr>
<tr>
<td>873</td>
<td>0.2883</td>
<td>0.2838</td>
</tr>
<tr>
<td>923</td>
<td>0.2874</td>
<td>0.2860</td>
</tr>
<tr>
<td>973</td>
<td>0.2872</td>
<td>0.2863</td>
</tr>
</tbody>
</table>
tetragonal lattice after tempering at 723 K (and even as high as 773 K). Above 773 K, the conventional specimens precipitate carbides and the $c/a$ ratio is decreased.

The $c/a$ ratios decreased with increasing tempering temperature in the cryogenic samples. The cryogenically treated specimens do not show the same behavior at 673 to 773 K as the conventional specimens because of the transformation from retained austenite to martensite produced by cryogenic treatment. Cryogenic treatment favors carbon clustering in martensite and transition carbide precipitation. This results in a more homogeneous and finer carbide distribution after tempering. The martensite $a$ and $c$ axes are only related to the carbon content in the steel. At tempering temperatures higher than 873 K, the body center tetragonal (BCT) structure slowly transforms into a body center cubic (BCC) structure, with the $a$ axis and $c$ axis lengths close to 0.2861 nm.

### 3.4 Dimensional changes

The martensitic transformation causes dimensional changes in the quenched specimens during tempering. Figures 8 and 9 show the dimensional change, retained austenite, and residual stress in the treated specimens as a function of the tempering temperature for both the conventional and cryogenic treatments, respectively. The cryogenic treatment is postulated to substantially convert retained austenite into martensite and produces a higher hardness compared with high-temperature tempering. The specimen length and diameter change ratio is determined by the value of the treated length (diameter) minus the original length (diameter) divided by the original length (diameter).

As can be seen in Figs. 8(a) and 8(b), the percent change in length (longitudinal direction) of the quenched specimen showed a positive value of 0.02% relative to the original ferrite matrix. When the quenched specimen was tempered at temperatures below 773 K, the percent change ratios decreased because of martensite contraction resulting from released trapped carbon. For the specimen tempered at 873 K, the length change ratio increased according to the austenite into martensite transformation; this was confirmed by the $c/a$ ratio enhancement indicated by the XRD results. The change...
in the diameter (transverse direction) of the specimens showed the same trends as that in the longitudinal direction as a function of the tempering temperature. The change in the transverse direction was greater than the change in the longitudinal direction.

Austenite and martensite lattice parameters were used to estimate the change in carbon content, which has a massive influence on secondary hardening. In the tempering process study, the amount of retained austenite in the specimens was tremendously reduced by tempering at temperatures higher than 723 K in conventionally treated specimens. The iron–carbon transitional carbide is precipitated at 473 to 573 K, and the alloy carbide for secondary hardening is precipitated at 473 to 573 K. These precipitation reactions cause a macroscopic size change in JIS SKD 11 steel and microscopic change in the c axis length in martensite. The size change and martensite lattice parameter c/a ratio are decreased by the martensite carbon concentration when the tempering temperature increases to 573 K. The transformation from retained austenite to martensite in high alloy steel is caused by tempering higher than 723 K without cryogenic treatment. The steel specimen size is expanded by the transformation of retained austenite into martensite. This data and the data for the corresponding change in c axis can be found in Table 2. In analyzing the data, the change ratio is influenced by both the amount of retained austenite and the martensite content. A significant correlation is found between the martensite lattice parameter and specimen size.

The dimensional changes in the cryogenic specimens as a function of the tempering temperature are shown in Figs. 9(a) and 9(b). The amount of retained austenite in as-quenched specimens after cryogenic treatment was reduced to only 3%. The specimen dimension change ratios were significantly decreased with the tempering temperature because the amount of retained austenite was reduced by cryogenic treatment. Tempering after cryogenic treatment contributes to the release of trapped carbon from the martensite. The present finding contributes to the understanding of the various forces acting on the martensite lattice parameters that are closely related to the overall size change and distortion.

3.5 Residual stresses

Steel deformation processes such as rolling produce overall shape changes accompanying grain shape changes and a large number of dislocations. The shapes of primary carbides in JIS SKD 11 tool steel adopt a linear structure in the longitudinal direction induced by the rolling process. The resulting stress distribution in the longitudinal and transverse directions in the surface regions is dependent upon the manufacturing process i.e., rolling contact loading. For the as-received JIS SKD 11 specimens, a tensile stress (180 MPa) in the longitudinal direction and a compressive stress (−176 MPa) in the transverse direction was obtained using PULSTEC μ-X360 portable X-ray residual stress analyzer using the cos α method with Cr radiation. The longitudinal residual tensile stresses were obtained in the rolling direction according to the carbide alignment and elongated ferrite grains. The compressive stresses in the specimen transverse direction resulted from the interaction with the die.

The residual stress distribution in the conventional specimen as function of tempering temperature is shown in Fig. 8. After quenching, the phase transformation from austenite to martensite occurs and the residual stress distribution in the quenched state differs from that in the as-received steel. The conventional sample in the as-quenched condition had a surface compressive residual stress of −118 and −85 MPa in the longitudinal and transverse directions, respectively. Because a redistribution of alloy elements and a change in elongated ferrite were produced by steel austenitizing, the stress distribution of the quenched specimens was uniform according to the large number of trapped carbon atoms in the martensite. When the quenched specimens were tempered, the compressive residual stress decreased because of the release of the trapped carbon atoms.

The amount of retained austenite exerts a significant influence both on the magnitude of the compressive stresses and on the dimensional stability. As can be seen in Fig. 8, the dimensional change and the magnitude of the residual stress are influenced by the amount of retained austenite after tempering at 773 K. As reported by Dommarto et al., the volumetric change produced during retained austenite decomposition contributes to the generation of compressive residual stresses. In summary, the transformation of retained austenite into martensite and the precipitation of alloy carbides lead to increased martensite lattice parameters and dimensional changes because of residual stresses. The residual stress is also changed to generate compressive residual stresses.

Figure 9 shows the residual stress distribution in the cryogenic specimens as a function of the tempering temperature. A large compressive stress (−324 MPa) is obtained in the specimen by using a 113 K cryogenic treatment. This result is in agreement with Bensley’s study, in which an increase in the compressive residual stress occurred in steel subjected to cryogenic treatment prior to tempering. The retained austenite in the quenched specimen was transformed into martensite by the cryogenic treatments. The compressive residual stress in the cryogenic treated specimens only decreased in magnitude because of the trapped carbon atoms released during tempering. The large relaxation in residual stress in the cryogenic specimens reflects the amount of trapped carbon released to form carbide precipitates during tempering. The hardness and dimension change ratio of the cryogenic tempered specimens decreased as tempering temperature was increased by the reduction in the amount of trapped carbon. Similarly, the magnitude of the compressive residual stress was gradually reduced to zero.

4. Conclusion

This study reports preliminary research on the residual stress and dimensional changes related to the lattice parameters in the JIS SKD 11 tool steel subjected to both conventional and cryogenic treatments. It finds that:

1. The grain boundaries of the primary austenite are easily identified in the quenched specimens using optical microscopy. When quenched specimens are tempered at 773 and 873 K, the grain boundaries of the tempered martensite matrix disappear.
From the XRD results of SKD 11 quenched and subsequently tempered specimens, retained austenite peaks were observed at low-temperature tempering (473 K), but no evidence of retained austenite peaks was observed at high tempering temperatures. There were also no obvious retained austenite peaks observed in the cryogenically treated specimen in the quenched and tempered states.

Trapped carbon atoms are released from the martensite, and the martensite c/a ratios and the dimensional changes in the conventionally treated specimens treated decrease before tempering at temperatures below 673 K. However, these values increase because of the associated increase in the c-axis when tempering is conducted at temperatures higher than 673 K. At that temperature, the retained austenite is converted into martensite.

The martensite c/a ratios and dimension change ratios of the cryogenically treated specimens were significantly decreased with tempering temperature because the retained austenite is reduced by cryogenic treatment.

For the conventionally treated specimens, when the quenched specimens were tempered, the compressive residual stress first decreased as a result of the release of trapped carbon atoms. However, the transformation of retained austenite into martensite and the precipitation of alloy carbides leads to an increase in the martensite lattice parameters, and dimensional change leads to residual stress. The residual stress is also changed to generate compressive residual stresses.

The hardness and dimension change ratio of the cryogenic specimen decreased as the tempering temperature was increased by the reduction in the amount of trapped carbon. Similarly, the magnitude of the compressive residual stress was reduced to zero.

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