Retained Austenite Analysis

Background

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 a 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.

<table>
<thead>
<tr>
<th>Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dimensional Control</td>
</tr>
<tr>
<td>High Speed Tool Steels</td>
</tr>
<tr>
<td>Forgings</td>
</tr>
<tr>
<td>Optimizing Heat Treatments</td>
</tr>
<tr>
<td>High Toughness Alloys</td>
</tr>
<tr>
<td>Bearings</td>
</tr>
<tr>
<td>Case Hardening</td>
</tr>
<tr>
<td>Failure Analysis</td>
</tr>
</tbody>
</table>

Analysis Method

Since Austenite has a different crystal structure from Martensite and the other forms of steel (Ferrite, Bainite and Pearlite), the resulting diffraction pattern will be different also. Thus, we can estimate the amount of Retained Austenite by comparing the intensities of diffraction peaks arising from each of the phases. In the absence of significant undissolved carbides and preferred orientation, there is a good correlation between the intensity ratio and the volume fraction of retained Austenite.

Two standards (ASTM E975 and SAE SP-453) for Austenite measurements are in common use. Both assume that the material has a nearly random orientation and has few carbides. The methods is illustrated in Figure 1, which compares the 200 Martensite (M200) peak with the

![Figure 1 Diffraction pattern used for retained Austenite analysis](image-url)
Problem Areas

Austenite 200 and 220 peaks (A200 & A220, respectively). In order to check for preferred orientation, the ratio of the integrated intensities of the two Austenite peaks is compared to the theoretical value of 1.475. If the ratio is between the limits of 1.2 to 1.8, then the sample is considered to be free of preferred orientation.

In order to determine the relative amounts of the Austenite and Martensite, a correction must be made for the differences in scattering power of the two phases. Since Austenite scatters X-rays better than Martensite, the intensity from the Austenite must be multiplied by 0.572 (for 200 reflection) or 0.388 (for 220 reflection) to account for this discrepancy. Once this is done, then the volume percent of retained Austenite is in direct proportion to its corrected intensity ratio.

For the example shown in Figure 1, the following intensities were collected:

- Austenite 200 2559
- Martensite 200 23892
- Austenite 220 3869

After the corrections are applied, the volume percent retained Austenite can be computed:

a) using Austenite 200 reflection:

\[
\text{vol}\% = \frac{(2559 \times 0.572)}{(2559 \times 0.572 + 23892)} = 5.77\%
\]

b) using the Austenite 220 reflection:

\[
\text{vol}\% = \frac{(3869 \times 0.388)}{(3869 \times 0.388 + 23892)} = 5.91\%
\]

Problem Areas

Two problem areas are likely to come up when doing retained Austenite analysis. The first arises when a significant amount of undissolved carbides is present, while the second occurs when either the Austenite or the Martensite has a preferred orientation.

In general, both problems are difficult to solve using conventional methods, such as those outlined in the ASTM and SAE standards. To overcome these problems, the preferred way today is to use the Rietveld whole pattern method whereby the entire diffraction pattern is analyzed instead of just the three peaks shown in Figure 1. The Rietveld refinement is a fundamental parameters method in which the diffraction patterns from each phase (with or without texture) is modeled and scaled to provide a least squares fit to the observed diffraction pattern.

With the simplified method where there are no carbides or preferred orientation, the ASTM/SAE methods are accurate to about 1% with a sensitivity level of about 0.5%. In the more complicated cases where the Rietveld method is required, similar results can be expected, even in the presence of carbides and texture.

Our Pledge

H & M Analytical Services has over 30 years experience in X-ray diffraction. With our state-of-the-art equipment, we will strive to apply our experience and knowledge to solve your most challenging problems. In most cases, we will provide turnaround of 24 hours on phase identification analyses at no additional cost. Sample preparation services are also available. Most samples can be run on a flat fee basis. For details, please contact us.

Other Services We Provide

We provide a wide range of X-ray diffraction services, including:

- residual stress analysis
- precision lattice parameter
- texture analysis
- phase identification
- Rietveld analysis
- particle size determination
- high temperature XRD
- analysis of modulated films
- misfit strains
- fiber analysis
- crystal orientation
- grazing incidence angle
- retained austenite analysis