Calculating Retained Austenite in Steel Post Magnetic Processing Using X-Ray Diffraction

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Nathan Pappas is a first year senior, double majoring in Mathematics Education and Mathematics Option 1. He plans to graduate in May 2008 and pursue a Ph.D. in mathematics. Nathan conducted the research for this article during an internship through the U.S. Department of Energy at Oak Ridge National Laboratory in the summer of 2006.

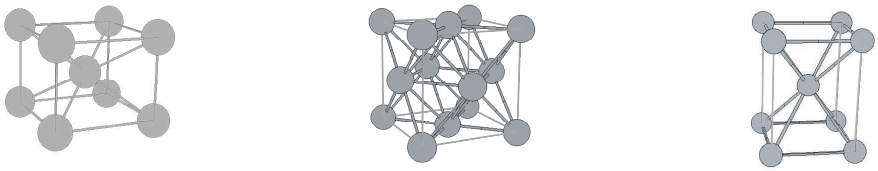
During the summer of 2006, I had the opportunity to participate in an internship through the U.S. Department of Energy (DOE) at Oak Ridge National Laboratory (ORNL). ORNL is the nations largest national laboratory that is funded by the DOE. ORNL is known best for developing the technology for the atomic bomb and was a classified area for many years. Today ORNL's research spans all areas of science from robotics and particle accelerators, to material science and hybrid lighting systems. I worked in the Materials Science and Technology Division with the Diffraction and Thermophysical Properties Group and was assigned to work on two projects under Dr. Thomas Watkins. The first project was to alter code for a residual stress program to accept new data files and the second was to calculate retained austenite in steel. I spent the majority of my time collecting and calculating data on sample of steel using x-ray diffraction. To understand the process of calculating retained austenite using x-ray diffraction, we must first know what austenite and x-ray diffraction are.

Background

Steel is an iron-carbon alloy that contains up to 2% carbon by weight and exists in three different phases as a solid [1]:

α - Fe	BCC	Ferrite	-273	to	912 °C
γ - Fe	FCC	Austenite	912	to	1394 °C
δ - Fe	BCC		1394	to	1538 °C

Ferrite has body center cubic (bcc) crystal structure, while austenite has face center cubic (fcc) crystal structure. Martensite, which will be discussed later, has body center tetragonal (bct) crystal structure. The only difference between bcc and bct is that bct is not a cube; it is stretched in the vertical



bcc fcc bct

Figure 1: crystal structures [8]

direction. The phases are physically different from each other in that the same atoms are arranged differently relative to each other in the crystal lattice. Steel is a polycrystalline material. That is, the atoms are arranged in a specific way relative to each other. In contrast, glass is not a crystalline material, which is important because it allows us to see through glass due to the atoms being randomly oriented relative to each other.

Understanding which phases of iron and carbon form as a function of temperature and weight percent carbon is important in order to process/manufacture steel with desired properties. Each phase has different characteristics, for instance strength, hardness, and ductility (how easily it can bend and not break). These properties are important when making steel. For example, steel that is used in a ball bearing needs different properties than steel used in a high-rise building. When steel is heated above 725 °C, austenite starts to form in steel [1]. Since austenite has fcc crystal structure, there is space for carbon atoms to diffuse into the structure without causing any structural change. When steel is in its austenite phase and quenched in a medium (usually water, oil, or gas) martensite forms due to the fact that the carbon atoms do not have time to diffuse out of the crystal structure [1]. The trapped carbon atoms result in the bct crystal structure rather than bcc.

During the manufacture of steel, the steel is usually heated to the austenitic forming region then quenched. Some of the austenite is retained in the steel at room temperature. Typically the steel is then heat treated at lower temperatures to allow some of the carbon to diffuse out of the bct martensite. This produces a steel, which is more ductile but also less hard. Any retained austenite is often undesired. "Since retained austenite can transform with a nominal 4% volume expansion into martensite, lower amounts of retained austenite are usually sought in order to avoid any corresponding distortion and/or loss of fracture toughness in the final piece" [6]. So retained austenite causes distortion in the steel, which for most precision pieces of steel, like for a ball bearing, would be unwanted. Heat-treating steel, as stated above, is usually timely and costly, so research has been conducted on alternate ways to reduce the amount of retained austenite. Research [2, 4, 5] has shown that the application of a high or ultra high magnetic field can alter the crystal structure and microstructure in medium and high carbon steels. The application of a magnetic field as a method for reducing the amount of retained austenite in steel is based on the thermodynamic argument that paramagnetic austenite is destabilized in the presence of a high to ultra high magnetic field [5].

Figure 2: Nathan Pappas using an x-ray diffractometer

Research Procedure

Research was conducted at Oak Ridge National Laboratory to investigate the effect of a magnetic field on SAE 52100 steel, which contains 1.45 wt% Cr and 1.0 wt% C [6]. During this research, I was responsible for collecting the data on the magnetically processed samples and calculating the retained austenite for all of the samples. To determine whether the magnetic field had any effect on the steel, x-ray diffraction was used to collect quantitative data corresponding to the amount of each phase (austenite and ferrite/martensite) present post processing. Jade software[1] was used in the phase identification analysis of the diffraction data. That is, the program identifies what peak corresponds to each phase, and uses least squares regression to fit curves to the data. Using the data collected in Jade, the amount of retained austenite was calculated using the American Society of Automotive Engineers (ASTM) standard for randomly oriented samples [7]. See [6] for experimental details and a full discussion of the results.

X-ray Diffraction

X-ray diffraction is a characterization method for crystalline materials, such as steel. Because x-rays have a wavelength comparable in size to atoms, the x-ray can penetrate a material and probe the arrangement of the atoms (for more detail, see [3, 9]). X-rays primarily interact with the electrons of the atoms and when scattering occurs, some photons from the incidence beam (x-ray beam) are deflected away from the direction in which they were traveling [9]. The x-rays that are measured during the scattering process are those in which the wavelength did not change. Since steel is a crystalline material, the atoms are arranged in a periodic fashion, i.e., planes of atoms. The diffracted waves result in sharp interference peaks that are shown in Figure 3. The peaks in a diffraction pattern are directly related to the interplanar distance, which is

[1] Jade 6.5-XRD Pattern Processing, MDI, Livermore, CA

why x-ray diffraction is used in the phase identification in steel. Remember, the different phases of iron have different crystal structures and thus different interplanar spacings.

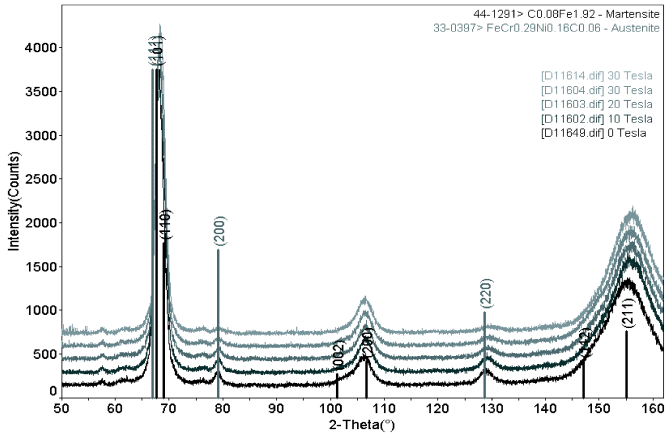


Figure 3: The XRD patterns of 52100 samples subjected to 0, 10, 20, and 30 T magnetic fields for 10 min immediately after quenching [6]

Bragg's Law states that by knowing the wavelength of the x-ray and the angle in which the beam is diffracting from the material, one can determine the atomic distance of a material. That is, Bragg's Law can be used to determine how far apart the atoms are from each other in the material. You probably never thought one of those silly trigonometric equations could tell you how far apart atoms are in a piece of metal did you? Specifically, we have

$$\lambda = 2d\sin\theta \qquad \text{[Bragg's Law]}, \tag{1}$$

where d is the interplanar distance, θ is the half angle of the Bragg or diffraction angle, and λ is the wavelength of the x-ray used [9].

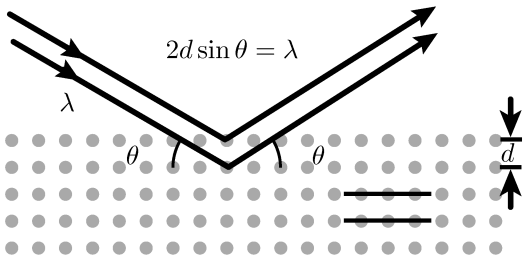


Figure 4: The spheres represent atoms in the crystal structure

Without going into detail, the sets of peaks in Figure 3 correspond to different phases in the samples. The relative area of the peaks is related to the

amount of each phase present within the depth of penetration of the x-rays (here $\sim 10\mu m$), which can be calculated.

Curve Fitting and Calculating

Jade is a software program that uses least squares regression to fit a curve to each peak in the diffraction pattern to determine the peak areas. In particular for the experiments at ORNL, a Pseudo-Voigt Profile Fit equation was used to complete the curve fitting [10]:

$$y = a_0 \left[(1 - a_3) \exp\left(-(\ln 2)(\frac{x - a_1}{a_2})^2 \right) + \frac{a_3}{1 + (\frac{x - a_1}{a_2})^2} \right] \tag{2}$$

where a_0 = peak maximum
a_1 = position of peak maximum (2-theta angle)
a_2 = full width of peak at half of the maximum (FWHM)
a_3 = proportion of the Gaussian and Larentzian peak widths

A Pseudo-Voigt function is the sum of a Gaussian and a Lorentzian function [10]. In Jade, the user selects a peak to be fitted. The program then iterates the function to minimize the error of the curve from the data. In Figure 5, the top line is the error of the fitted curves to the data.

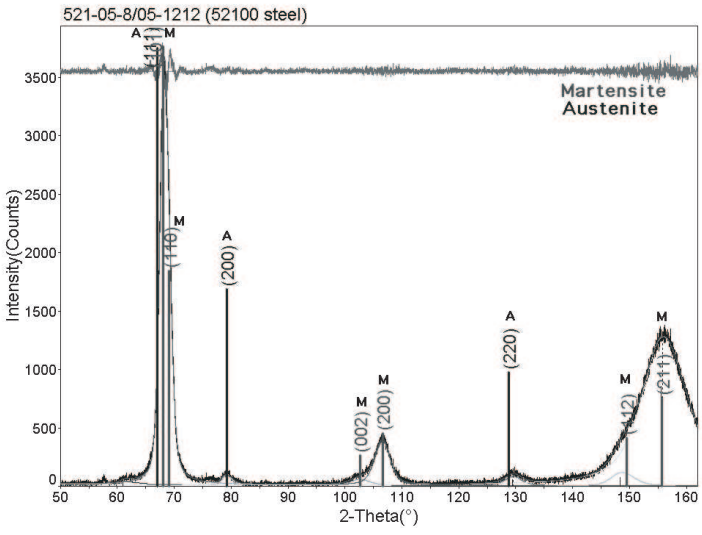


Figure 5: Jade Profile Fit of Diffraction Pattern

Once the pattern has been curve fit, Jade then integrates the curves to find the area underneath each peak. This total area for a set of peaks corresponds to the integrated intensity of each phase present in the material.

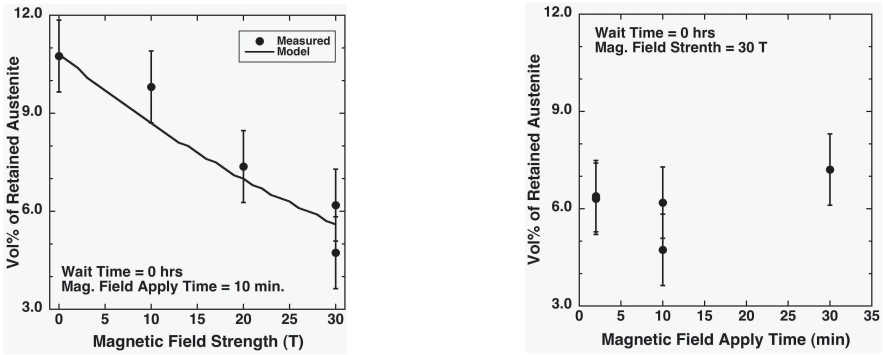


Figure 6: The volume fraction of retained austenite as a function of magnetic field strength (left) and as a function of magnetic field apply time (right)

The amount of retained austenite was calculated using the ASTM standard for randomly oriented samples [7] where R is a scale factor associated with phases and materials used, I is the calculated integrated intensity total for a phase, and V is the volume fraction of the phase. The austenite phase is represented by γ and the ferrite/martensite phase is represented by α.

$$\% \text{ of } RA = (V_\gamma)100 = \left(\frac{\frac{I_\gamma}{R_\gamma}}{\frac{I_\gamma}{R_\gamma} + \frac{I_\alpha}{R_\alpha}} \right) 100 \tag{3}$$

Results

The results of the experiment showed that retained austenite could be lowered by means of a magnetic field if applied shortly after the quench. The (200) peak that is shown in Figure 3 reflects the fcc structure of the steel. That is, peak (200) reflects austenite. It is clear from the figure that the peak is decreasing in size as the intensity of field increases. Thus the diffraction pattern visually shows the decrease in retained austenite, as does Figure 6 (left). The research also showed that it is not how long the field is applied for, but rather the intensity of the field that is important. This is shown in Figure 6 (right). It is clear from Figure 6 (right) that the amount of retained austenite is independent of the magnetic field apply time. For a full discussion of the results, see [6].

Conclusion

As seen in calculating retained austenite, material science uses multiple branches of mathematics. Nonlinear regression, integration, trigonometry, and arithmetic were all used in calculating the amount of retained austenite. During my internship at ORNL I did not learn any new mathematics besides the Pseudo-Voigt equation, but I did learn where the mathematics I had learned at Ball

State is used in cutting edge research. Mathematics can be applied to many disciplines as was shown in this application to material science. It has been said that engineering is applied physics, and physics is applied mathematics. Clearly, mathematics is fundamental to the sciences.

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