

Neutron Diffraction Studies of Intercritically Austempered Ductile Irons

2011-01-0033

Published
04/12/2011

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doi:[10.4271/2011-01-0033](https://doi.org/10.4271/2011-01-0033)

ABSTRACT

Neutron diffraction is a powerful tool that can be used to identify the phases present and to measure the spacing of the atomic planes in a material. Thus, the residual stresses can be determined within a component and/or the phases present. New intercritically austempered irons rely on the unique properties of the austenite phase present in their microstructures. If these materials are to see widespread use, methods to verify the quality (behavior consistency) of these materials and to provide guidance for further optimization will be needed. Neutron diffraction studies were performed at the second generation neutron residual stress facility (NRSF2) at the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory on a variety of intercritically austempered irons. For similar materials, such as TRIP steels, the strengthening mechanism involves the transformation of metastable austenite to martensite during deformation. For the intercritically austempered ductile irons two different deformation/strengthening mechanisms, phase transformation and slip, dependent upon the iron chemistry, were observed. Lattice strain and phase fraction data as a function of applied stress are presented.

INTRODUCTION

Intercritically austempered ductile irons have microstructures similar to the Advanced High Strength Steels (AHSS), which are gaining increased use in automotive applications since they can: 1) reduce vehicle weight and therefore improve vehicle fuel economy and 2) improve vehicle safety by increasing the vehicle structure's ability to safely absorb energy in a crash. Producing similar properties in ductile iron

would allow for additional weight reduction opportunities and offer a lower cost alternative in applications such as crankshafts and chassis safety components [1, 2, 3, 4, 5].

Intercritically austempered ductile irons have a desirable combination of high tensile strength and ductility, high fatigue resistance and good machinability due to a microstructure consisting of well-formed graphite nodules in a matrix of ferrite plus islands of austenite, [Figure 1](#). The continuous ferrite matrix provides good tensile strength, high ductility and good machinability, and the austenite particles significantly increase tensile strength and fatigue strength. Previously, it was believed that the strengthening mechanism was transformation induced plasticity (TRIP), i.e., energy is absorbed when the austenite transforms to martensite, which is the strengthening mechanism in AHSS [6,7]. This study will show that there is more than one possible strengthening mechanism and that one strengthening mechanism is controlled by the material chemistry.

In order to further the development of these materials, neutron diffraction was used to study the deformation mechanisms in intercritically austempered ductile irons. Neutron diffraction is a powerful tool that can be used to determine residual stresses, changes in lattice parameter and phases present within a volume of material, such as the entire cross section of a tensile specimen. The purpose of this research was twofold: (1) to apply neutron diffraction techniques to determine the strengthening mechanisms in intercritically austempered ductile irons of different chemistries and (2) to determine if chemistry had an influence on these mechanisms. This information would

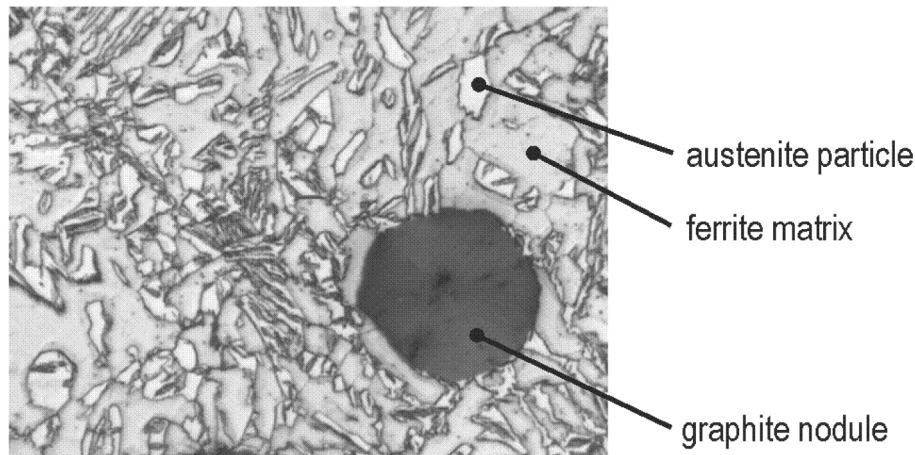


Figure 1. Typical microstructure of an intercritically austempered ductile iron.

allow the optimization of the material chemistry resulting in the best combination of properties at the lowest possible cost.

POLYCRYSTALLINE DIFFRACTION (IN BRIEF)

Diffraction studies can determine crystallinity (are “peaks” present), the pattern of peaks identifies the crystalline phase(s) present (every phase has a distinct set of peaks), the number of different crystalline phases present (requires sorting out which peak belongs to which phase), the amount of each crystalline phase present (quantitative phase analysis), the size of the diffracting domains (“crystallite” size) and the strains in the crystal structure.

Simplistically, a diffraction peak has three main descriptors: angular position, height or intensity and width or breadth. Often, curve fitting is used to mathematically determine the angular position, maximum intensity, integrated intensity (area under the peak) and peak width (often the full width at half maximum intensity, FWHM) from the experimental data. The peak fitting functions typically used are Gauss, Lorentz, Pseudo-Voigt or Pearson-VII. Peaks that overlap can be a challenge to properly separate. For a given wavelength, the angular position of a peak (2θ) is determined by the spacing of the atomic planes. The peak positions in the pattern are used for identifying a phase while the relative peak intensities of a phase provide a measure of the randomness, or not (called “preferred” orientation or crystallographic texture) of the diffracting domains. The relative integrated intensity under the diffraction peaks can be used to determine the amount of each phase present in a mixture of phases. The width or breadth of a peak can be used to determine the average size of the crystallites and to determine if defects and/or gradients are present. The size of the individual crystallites (grains) can cause significant peak broadening when the crystallite size is less than about 100 nm. Broadening may also originate from gradients or wide

distributions of non-uniform strains, defects, stacking faults, chemistry, disorder, etc. [8] It should be noted that uniform (within the diffracting volume) strain causes the location of a peak to shift, and are sometimes referred to as macro- or type I strains extending over mm length scale. Today, micro- or type II strains typically refer to grain-to-grain strains or strains extending over μm length scale. Non-uniform strains extend over the nm length scale and have been referred to as root mean square strains (rms) as well as microstrains in literature pre ~1980. [9]

X-rays are commonly used in diffraction work and are electromagnetic waves that interact with the electron clouds of an atom. Electron clouds are not points in space but have a finite area, which is about the same magnitude as X-ray wavelengths. Since electron clouds are not points in space but are “spreadout” over an area, their scattering power varies with the angle of observation, 2θ . As a consequence of this effect, the X-ray diffraction peaks at higher angles have lower intensities compared to those at lower angles.

Neutrons may also be used in diffraction work and are particle waves that interact with the nucleus of an atom. Therefore, the scattering power of neutrons is independent of the angular position, 2θ . Neutrons are produced by either fission (heavy elements breaking into lighter elements, which liberates neutrons) or by bombarding protons impinging on a nuclei that then releases “spallation” neutrons. The energy of fission neutrons (~ 1 MeV) is too high for practical diffraction experiments so they are slowed down (moderated). Hot neutrons have wavelengths of 0.03 - 0.1 nm, thermal neutrons have wavelengths of 0.1-0.4 nm and cold neutrons have wavelengths of < 3 nm.

Comparison of X-ray and neutron diffraction patterns taken at the same wavelength reveals the peaks would be in the same positions but the relative intensities would be different. Advantages of neutrons compared to X-rays are lower

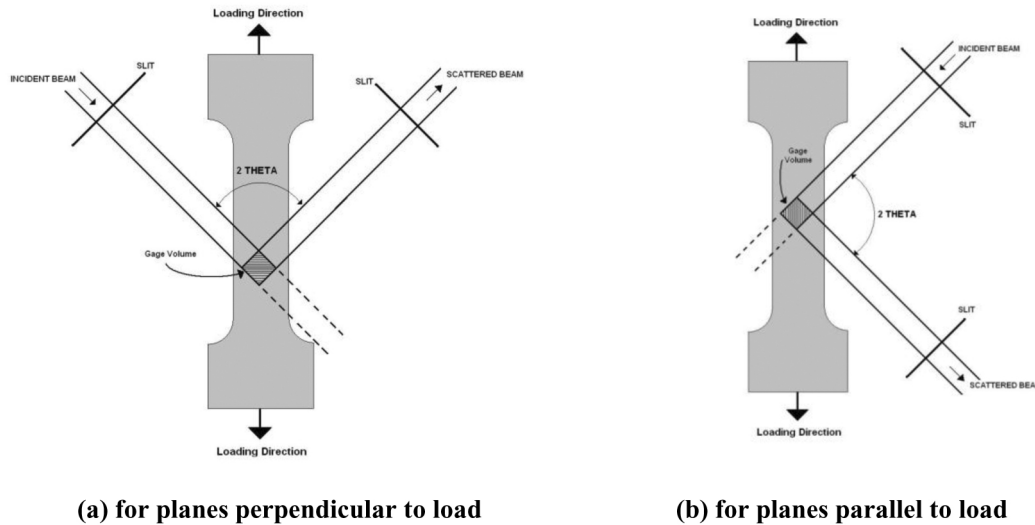


Figure 2. Schematic of neutron diffraction set up showing the slits relative to the tensile sample. Orientation (a) would measure the interplanar spacings of the planes perpendicular to the loading direction and orientation (b) would measure the interplanar spacings of the planes parallel to the loading direction.

absorption so they penetrate deeper and provide the ability to analyze the bulk, discriminate between neighboring elements and isotopes, detect light elements and investigate magnetic structures. Also, the relative intensities of high angle Bragg reflections are stronger for neutrons compared to X-rays. However, neutron sources have limited availability and produce beams of lower flux than X-ray sources. Neutrons can also activate some samples. Peak resolution on neutron instruments is generally poor relative to standard x-ray instruments. Materials with boron, ^{11}B , and hydrogen, ^1H , strongly absorb neutrons and hydrogen containing materials have very high backgrounds.

In this study, neutron diffraction was used for strain/stress measurement. The interplanar spacings or lattice spacings, d_{hkl} , are related to wavelength, λ , and the scattering angle, 2θ , by Bragg's law:[10]

$$\lambda = 2 d_{hkl} \sin\theta \quad (1)$$

The elastic strain, ϵ_{hkl} , can be determined in a sample using the following equation:

$$\epsilon_{hkl} = (d_{hkl} - d_{hkl}^0) / d_{hkl}^0 \quad (2)$$

where the subscript hkl refers to a particular set of crystallographic plane(s) measured and d_{hkl}^0 is the interplanar spacing with zero strain. If the appropriate elastic constants are known (preferably plane specific (hkl) diffraction elastic constant), the applied or residual stress can be calculated from the strains measured in three orthogonal directions. It should be noted that only elastic strains can be measured

using diffraction; plastic strains cannot be measured using diffraction.

Since neutrons can penetrate metals to a significant depth (tens of millimeters [11]), it is possible to analyze specific volumes of material at fixed locations within the bulk of the specimen, which allows the non-destructive determination of 3-D residual maps. The size and shape of incident and diffracted neutron beams are defined by slits of absorbing material (often cadmium). These slits are typically between 0.5 and 5.0 mm wide. The volume of material analyzed in a sample is the intersection of the incident and diffracted beams. Schematic drawings showing neutron diffraction of a volume of material within a stressed sample are shown in Figure 2. The direction of the strain measurement is the bisector of the incident and diffracted beams called the diffraction vector. In a polycrystalline material, only the crystallites, often referred to as grains, that are oriented such that the plane normals are parallel to the diffraction vector will be detected. Each distinct grain will generate a distinct peak; the signals from all of the diffracting grains of a particular experimental orientation are summed into one peak yielding an average.

EXPERIMENTAL METHODS

Intercritically Austempered Ductile Iron (IADI). Ductile irons containing either nickel or manganese as the primary alloying elements were melted and cast at the University of Alabama at Birmingham casting lab. The cast alloys were austenitized at 740 or 760°C for 4 hours and then austempered at 357°C for 2.25 hours at a commercial heat treater. Two types of tensile specimens were needed for this study. Small-size tensile specimens, 9 mm diameter × 36 mm