

## **DIFFRACTING PARTICLE SIZE ANALYSIS OF MARTENSITE - RETAINED AUSTENITE MICROSTRUCTURES.**

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### **ABSTRACT**

Case carburized steels containing a martensite-retained austenite microstructure are typically analyzed for the amount of each phase using x-ray diffraction techniques which are well established. In addition, however, analysis of the diffracting particle size of both the retained austenite and the martensite provides additional detailed information about the microstructure. The diffracting particle size of the martensite was measured as a function of depth of four end-quench specimens (Jominy bars) ranging from 0.22 to 0.83 weight percent carbon. In addition, diffracting particle size of 53 case carburized samples was measured at a constant carbon composition, as a function of retained austenite content. Analysis of the data indicates that the diffracting particle size of the austenite becomes refined as the amount of martensite transforming from the austenite increases. This effect is due to the austenite being cut by the martensite and plastic deformation of the austenite by the martensite at lower temperatures. The martensite diffracting particle size remains relatively constant during transformation at a given carbon level, regardless of the degree of transformation. The martensite diffracting particle size does, however, increase as the morphology changes to a diffusion controlled transformation.

### **INTRODUCTION**

Engineered components and structures which utilize steel as a material along with a quench and temper heat treatment will experience different cooling rates as a function of location during the quenching process. In addition, the product may have a variable carbon content as a function of location, due to processes such as carburization or decarburization of the surface of the component. The two phases which are most prevalent in this type of microstructures include martensite and retained austenite. While the amount of each phase is of great importance and is certainly the parameter which is most often measured using x-ray diffraction, the diffracting particle size of both the martensite and retained austenite as a function of carbon content and cooling rate are also of interest and can be determined at the same time generally using the same data. If the diffracting particle size can be correlated with the cooling rate and carbon content, it can be used as a method for checking the consistency of a particular heat treatment process, or to determine information about the processing parameters used for a particular heat treatment operation.

## EXPERIMENTAL PROCEDURE

### End-Quench Test

The specimens studied were standard Jominy bars machined from symmetrical positions of a single AISI-SAE 4320H grade steel bar, carburized, and then homogenized which resulted in carbon levels of 0.22, 0.34, 0.46, and 0.84%. Chemistry of the starting material is listed in Table 1. These four carbon levels were achieved by carburizing the specimens 0, 20, 40, and 120 hours respectively. Homogenization was carried out at 1200°C for 192 hours in a neutral atmosphere of argon. The grain size was then refined by two thermal cycling treatments after of the homogenization treatments were employed.

Element	Weight %
Nickel	1.69%
Manganese	0.61%
Chromium	0.57%
Molybdenum	0.23%
Silicon	0.30%
Carbon	0.22%
Phosphorous	0.021%
Sulphur	0.020%
Copper	0.14%
Aluminum	0.05%

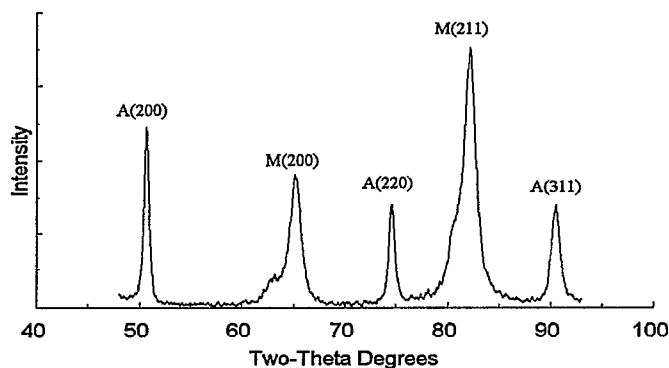
**Table 1:** Chemical analysis of the 4320H steel used for this investigation.

The Jominy bars were austenitized at 857°C, held at temperature for 40 minutes, and then end-quenched as per ASTM A255. [1] Water temperature was 16°C. Flats were ground on the longitudinal sides of the Jominy bars and Rockwell hardness profiles were performed.

X-ray diffraction measurements were made by grinding to several depths from the quenched end of the four Jominy bars. Surface roughness after grinding measured less than 0.8 microns. The surface damaged layer was then removed by electropolishing at 25 to 35 volts for 120 seconds in a 6% perchloric, 35% butoxyethanol, 59% methanol solution cooled by an ice water bath. This procedure removed eight to 16 µm of material. Finally, the electropolished surface was inspected using an optical microscope to ensure a uniform removal of material.

A Philips x-ray diffractometer was used with a copper anode tube powered at 40 kV and 30 mA. The diffractometer was outfitted with a graphite diffracted beam monochromator and a variable divergent slit. Since the cooling rate is a function of distance from the quenched end, copper  $k_{\alpha}$  radiation was selected to minimize x-ray penetration depth. This decreased the range of cooling rates averaged into each x-ray measurement.

Higher carbon content in the steel increases the tetragonality of the as-quenched martensite unit cell. This is clearly seen in the x-ray diffraction pattern from the as-



**Figure 1:** X-ray diffraction patterns of as-quenched 0.84% carbon steel.

quenched 0.84% carbon specimen in Figure 1. Because the amount of tetragonality depends on several factors such as carbon content and thermal history, the diffraction profile is affected to varying degrees. Although inexact, an assumption was made that the diffraction profiles are symmetrical, therefore a method of reflecting the high angle side data to the low side was used, thus removing the asymmetric effect of the tetragonality on the diffraction profile.

A single-profile Fourier analysis technique was used to determine the diffracting particle size<sup>[2]</sup>. This technique employs the Stokes correction for instrumental broadening.<sup>[3]</sup> Profiles from the NIST 660 LaB<sub>6</sub> standard reference material provide the instrument broadening function. The effective particle size was determined from the martensite M(200) reflection. Ferrous martensite contains very high dislocation densities in the order of 10<sup>11</sup> to 10<sup>12</sup> per cm<sup>2</sup>.<sup>[4]</sup> With this heavy defect structure of the martensite, the x-ray line broadening is predominantly produced by small coherent particles.

### Experimental Case Carburized Specimens

Fifty three 4320H steel specimens were case carburized with a 1.0% carbon potential gas, cooled to room temperature then austenitized, followed by an oil quench. Finally, the specimens were tempered at 176°C for three hours. The processing parameters such as austenitizing temperature, quench temperature, etc., were varied slightly to produce retained austenite contents ranging from 17 to 31%. Using a surface grinder, 500 μm of material was removed from the surface, then electropolished to remove the effects of grinding. Diffracting particle size was calculated from the retained austenite A(200) and martensite M(200) reflections. Because these specimens were tempered no tetragonality of the unit cell was present, therefore, it was not necessary to symmetrize the diffraction peak.

Retained austenite measurements were accomplished employing a four peak analysis method. The integrated intensities of two retained austenite, A(200) & A(220) and two martensite, M(200) & M(211) diffraction peaks were used to calculate the retained austenite content.<sup>[5]</sup> These peaks are indexed and shown in Figure 1.

## RESULTS AND DISCUSSION

### End-Quench Test

Rockwell C hardness of the Jominy bars as a function of distance from the quenched end are shown in Figure 2.

As expected, hardness increases with increasing carbon content. The lower carbon specimens exhibit decreased hardenability as compared to the higher carbon specimens. Both the 0.46% and 0.83% C specimens are through-hardened, maintaining their hardness 50 mm from the quenched end. Hardness of the 0.22 % C specimen

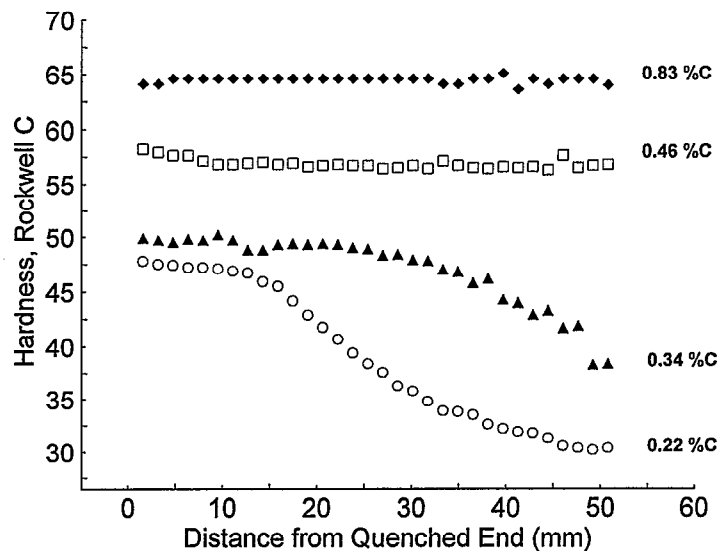


Figure 2: Rockwell C hardness profile of the four end-quenched specimens.

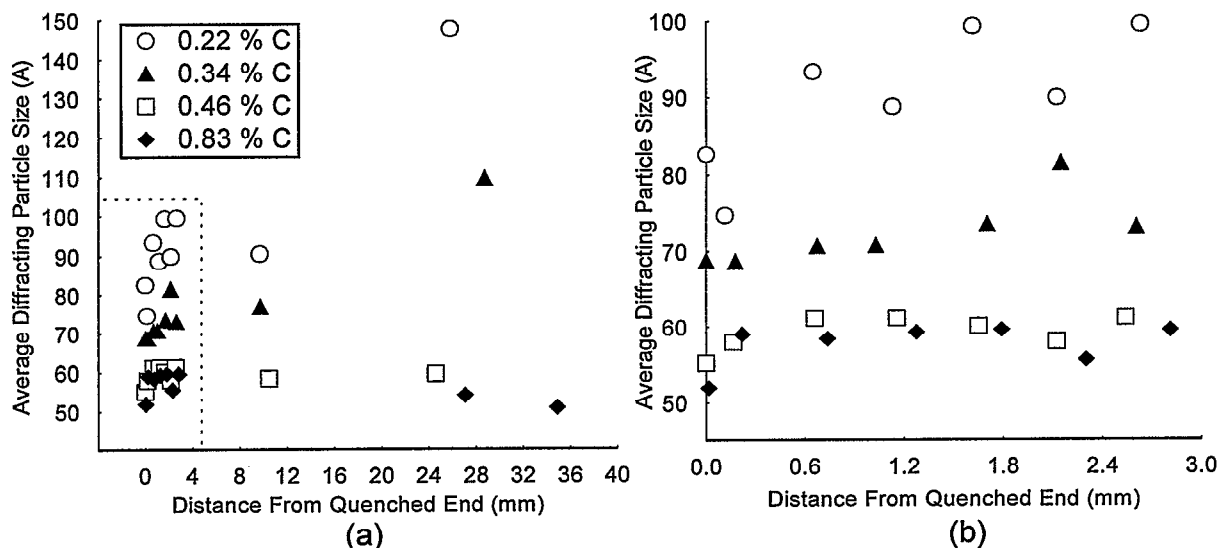
begins to decrease at a depth 14 mm from the quenched surface, while the 0.34% C specimen begins to decrease at 25 mm.

The diffracting particle size of the martensite as a function of distance from the quenched end for all carbon levels are shown in Figure 3a. Figure 3b is enlarged area from Figure 3a. An overall decrease in diffracting particle size is observed with increasing carbon content; however, very little difference between the 0.46% and 0.83% carbon specimens is observed. For all specimens, there is a sharp decrease in the diffracting particle size in the first couple of millimeters towards the quenched end. This decrease in diffracting particle size approaching the quenched end is due to a faster cooling rate effect. These faster cooling rates increase the amount of undercooling which occurs before transformation. Consequently, this increases the number of active sites for martensite nucleation. It is reasonable to expect that the length of crystal coherence becomes smaller as the density of martensite nucleation sites increases.

The martensite start temperature,  $M_s$ , may be calculated for low alloy steels by the empirical equation;

$$M_s (\text{°C}) = 500 - 333 C - 34 \text{ Mn} - 35 \text{ V} - 20 \text{ Cr} - 17 \text{ Ni} - 11 \text{ Mo} - 10 \text{ Cu} - 5 \text{ W} - 15 \text{ Co} + 30 \text{ Al}$$

or for these specimens,  $M_s (\text{°C}) = 437 - 333 C$ , where the alloying elements are given as weight percentages.<sup>[6]</sup> The  $M_s$  temperature decreases from approximately 363°C for the 0.22% C to 157°C for the 0.84% C specimens. The amount of undercooling created by the quench with 16°C water is greater in the lower carbon specimens when the  $M_s$  temperature is highest. This combination of high  $M_s$  temperature and positions near the quenched end where the cooling rate is faster produce the largest difference in diffracting particle size.

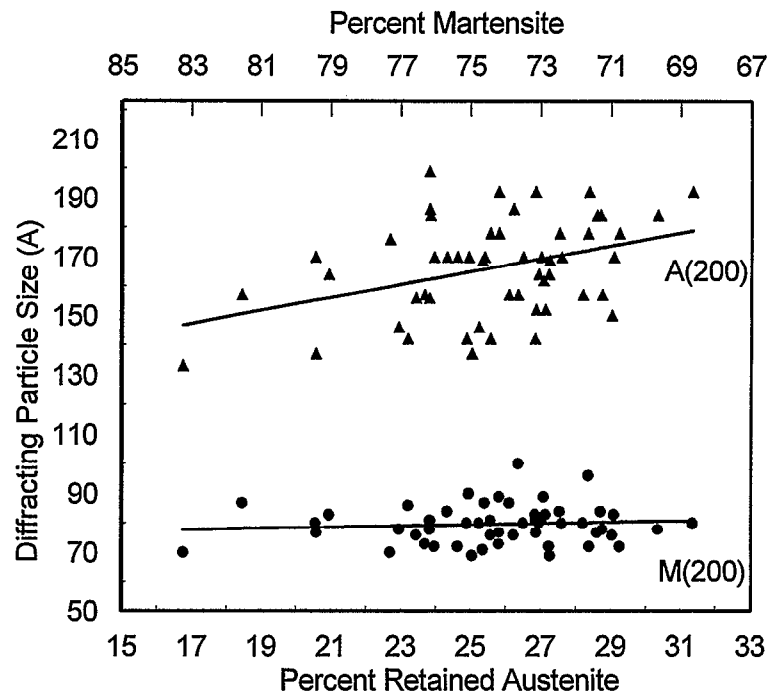


**Figure 3:** (a) Diffracting particle size measured from the martensite (200) as a function of distance from the quenched end. (b) Enlarged area from outlined in Figure 3(a).

An increase in the martensite diffracting particle size occurs where diffusion controlled transformation of austenite begins. [7] This is observed in the lower carbon specimens at depths which coincide with a decrease in hardness. No increase in martensite diffracting particle size is observed in the through hardened, higher carbon, specimens at depths up to 50 mm.

### Experimental Case Carburized Specimens

Diffracting particle size of the austenite and martensite phases is plotted as a function of retained austenite content in Figure 4. The diffracting particle size of the austenite phase remains constant as a function of retained austenite content. However, the retained austenite diffracting particle size decreases with increasing amount of martensite formed. This decrease is considered to be due to two effects. First, the increase amount of plastic deformation of the austenite as larger amounts of martensite is formed. Second, cutting of the austenite by the martensite as it forms.



**Figure 4:** Diffracting particle size of retained austenite and martensite as a function of percent retained austenite for the experimental case carburized specimens.

## CONCLUSIONS

From the results of this investigation the following conclusions can be made:

### End-quench Test

The martensite diffracting particle size decreases with increasing carbon content and with increasing cooling rate. A large increase in diffracting particle size is observed where diffusion controlled martensitic transformations begin to occur.

### Experimental Case Carburized Specimens

**Martensite:** Diffracting particle size remains constant with increasing amounts of retained austenite. Scatter of data are indicative of processing control.

**Retained Austenite:** Diffracting particle decreases with increasing amount of martensite. This is an indication of austenite particle cutting by the martensite plates during transformation and the increasing plastic deformation in the austenite due to larger amount of martensite formed.

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