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Rapid determination of the deformation-induced martensite in metastable stainless steels

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Determination of the amount of strain-induced martensite in metastable 18/8 stainless steel by X-ray diffractometry and quantitative metallography was examined. The need to use at least the three reflections of lowest order to include the significant components of texture, and the inaccuracy of quantitative metallography for determining the amount of martensite present, reported by previous workers, were confirmed. A simple magnetic method employing an analytical balance to measure the force required to break contact between the specimen surface and a hemispherical probe, calibrated for martensite contents up to 95%, provided a simple, rapid and reproducible means of determining the amount of α' (magnetic) martensite present. No non-magnetic ϵ martensite was detected by X-ray diffractometry.

1. Introduction

1.1. Techniques for determining martensite in metastable autenitic stainless steels

A rapid, simple and inexpensive method of determining the proportions of austenite and martensite greatly facilitates the study of deformation-induced transformation in metastable stainless steels, especially when it is directed toward a technological application, such as determining maximum elongation temperature and its dependence on stress system and deformation rate or other process variables [1].

X-ray diffraction and the Sucksmith magnetic balance [2–4], the Null Coil Magnetometer [5] and the magnetic induction method [6, 7] have been used for determining the martensite content of stainless steel specimens. Quantitative metallographic methods are time consuming and the morphology of martensite militates against easy measurement. Maxwell, Goldberg and Shyne [8] found that the martensite plates occuring in sheaves or elongated clusters of laths were too small to be resolved individually. As a result, optical microscopy indicated greater amounts of strain-induced martensite than actually existed.

When a test-piece is required for further deformation, it is impracticable to cut from the test-piece the small samples required for the Sucksmith magnetic balance and for instruments depending on induction (the use of which may also be relatively time-consuming).

The X-ray diffraction technique, although potentially the most accurate and suitable for definitive measurement and calibration of other methods, is the most time-consuming and usually requires that samples be cut for analysis. Its application has been described by a number of workers [9–13].

Durnin and Ridal [14] studied material with only a relatively small degree of preferred orientation, whilst others [15, 16] used reflections from only one austenite and one martensite plane. However, Miller [17] has shown that errors in analysis using X-rays can occur because of preferred orientation effects, and suggested that use of the integrated intensity from the austenite peaks would average out any preferred orientation which may be present in the austenite. Dickson [18] has extended the method to correct for modifications to peak intensities caused by extremely

high degrees of preferred orientation.

$$\frac{C_\gamma}{C_\alpha} = \frac{(I/R)\,2\,0\,0_\gamma + (I/R)\,2\,2\,0_\gamma + (I/R)\,3\,1\,1_\gamma}{(I/R)\,2\,0\,0_\alpha + (I/R)\,2\,1\,1_\alpha + (I/R)\,3\,1\,0_\alpha},$$

$$C_\alpha = \frac{1}{1 + C_\gamma/C_\alpha},$$

where C_γ is volume fraction of austenite phase, C_α is volume fracion of martensite phase, I is measured integrated intensity, and R is a factor depending on the crystal structure of the specimen and the Bragg angle θ (see Table I).

TABLE I Parameters required for calculating volume fraction of martensite using Mo$K\alpha$ radiation by Dickson's integrated intensity method

Reflections	Bragg Angle (θ)	Factor, R
$2\,0\,0_\gamma$	11.39	481
$2\,2\,0_\gamma$	16.32	298
$3\,1\,1_\gamma$	19.21	314
$2\,0\,0_\alpha$	14.35	224
$2\,1\,1_\alpha$	17.73	413
$3\,1\,0_\alpha$	22.98	132

Dickson showed that large errors arose when only a small number of reflections were considered. Generally, it is necessary to use a sufficient number of reflections from martensite and austenite planes to include all major components of the texture. Using reflections from two austenite and two martensite planes in the 18 wt% Cr– 8 wt% Ni austenitic stainless steel studied, the amount of martensite measured was 98% compared with 87.6 and 86.3% respectively for reflections from three austenite/three martensite and seven austenite/seven martensite planes.

The limit of accuracy of the method was given as ±2%, which accords with the work of Rosen *et al.* [19] who found the accuracy of the X-ray method to be ±1%.

1.2. The scope of the present work

A method was sought which would be suitable for determining the amount of martensite induced in stainless steel sheet deformed under the various stress systems (tension, tension/tension and tension/compression) which may arise in technological sheet forming processes, to permit determination of the appropriate maximum elongation temperature.

The use of a simple magnetic balance (described below) and X-ray and metallographic techniques for calibrating it were investigated.

2. Experimental

2.1. Preparation of Specimens

A number of tensile test specimens of 1 mm 18/8 stainless steel sheet of Type 301, prepared according to BS 18 : 1962, were strained in uniaxial tension at room temperature to various elongations at intervals of approximately 5% in the range 0 to 40%, a range which was expected to cover the amounts of martensite to be encountered during investigation of warm deep drawing (at the maximum elongation temperature).

The edges of all test specimens were removed by careful wet grinding and the surfaces were prepared by a repeated polish-and-etch technique, with a final light etch as described below to minimize any effects of transformation to austenite caused by the stamping and grinding operations.

2.2. Metallographic determination of martensite

Mounted specimens were ground wet on silicon carbide paper down to 600 grade, polished successively on 3 to 6 μm and $\frac{1}{2}$ to 1 μm diamond paste, and etched in an aqueous solution of 2.5 g $CuCl_2$, 2.5 g $FeCl_3$, 10 ml HNO_3, 50 ml HCl per 100 ml. Results were unsatisfactory because strain markings which appeared similar to martensite obscured the phase structure, and made impossible measurement of the amount of martensite (by the intercept method).

2.3. Martensite determination by X- radiographic analysis

A wide-angle goniometer mounted on a Philips 1.6 kW X-ray diffractometer producing Mo$K\alpha$ radiation was used. Continuous scans, to locate the appropriate peaks, were performed over a sufficiently wide angle to include background radiation, as well as that associated with reflecting planes, and for long enough to provide a satisfactory count of the background radiation. Martensite in the deformed specimens was determined respectively using the (2 0 0), (2 2 0) and (3 1 1) reflections for austenite and the (2 0 0), (2 1 1) and (3 1 0) reflections for α' martensite.

2.4. The magnetic balance

2.4.1. Description

The balance, illustrated in Fig. 1, consisted of a rod 30 mm long by 5 mm diameter of fully magnetised Alnico 5B, jacketed in a mild steel sheath designed using data provided by the manufacturers

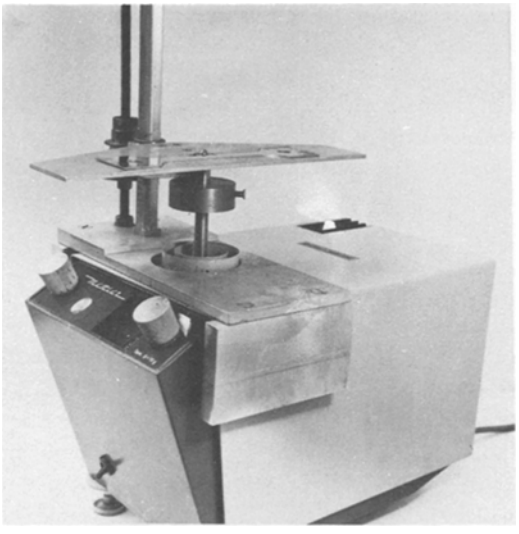

Figure 1 The magnetic balance

[20] to minimize flux leakage, capped on the exposed end by a mild steel hemisphere attached with Eastman 910 pressure-sensitive adhesive, and mounted in a brass holder which replaced the pan of a 0 to 3000 g Mettler analytical balance. The hemispherical contact minimized sensitivity to the size of the specimen and to roughness and curvature of its surface. Specimens of 1 mm stainless steel of Grade 301, deformed to induce degrees of transformation up to 95%, previously analysed for martensite by X-ray diffractometry, were clamped over a slot in a brass platform mounted on a rack which was raised until contact was broken, and the corresponding force of attraction was measured.

2.4.2. Calibration

Each point shown in Fig. 2 is the mean of five readings, taken at intervals of approximately 2.5 mm along the gauge length of a specimen subjected to deformation in simple tension, but the mode of deformation of the calibration specimen, (tensile, bulge or cup test), appeared to be of no significance. The variation between readings did not exceed 5%.

Use of only one X-ray reflection lead consistently to a higher estimate of the proportion of martensite present in all samples (Fig.2), whether they were almost completely martensitic or almost completely austenitic. In particular, it indicated that there was 13% martensite present in the unstrained condition, but none was detected by the magnetic balance. This over-estimate accords quantitatively with the finds of Dickson [18] in his investigation described previously, in which he found that use of two and three reflections led to estimated martensite contents of 98 and 87.6%, respectively, in material in which the definitive X-ray analysis using seven reflections indicated 86.3% martensite.

Although a number of previous investigators [3, 21, 22] have reported the presence of the (non-magnetic) hexagonal ϵ martensite in metastable stainless steels deformed in tension, only austenite and the (magnetic) α' martensite were detected in the present investigation. Generally, ϵ martensite has only been reported after deformation at, and below, room temperature, in amounts which decreased as the strain increased so that it is unlikely to have been necessary to

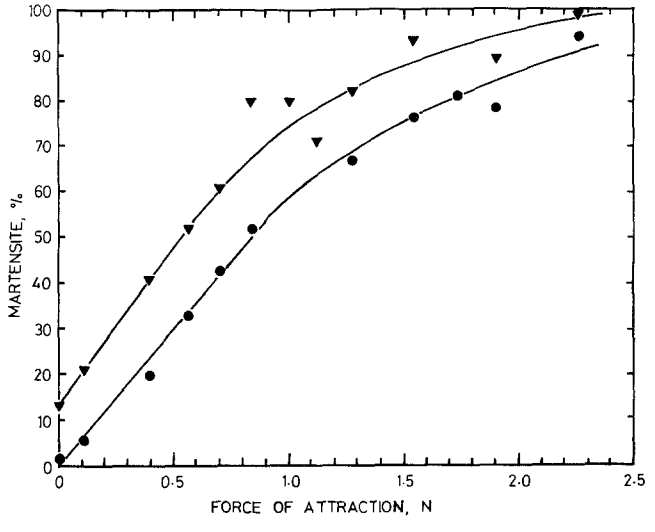

Figure 2 Calibration of the magnetic balance; Values obtained from the first austenite and the first martensite reflection ▼, values obtained from the first three austenite and the first three martensite reflections ●.

consider its presence as a source of error or complication in the work reported here.

2.4.3. Effect of variables on the calibration

Although the magnetic balance appears to provide a rapid, reproducible and reliable measure of the martensite content of the stainless steel sheet investigated, the extent to which composition and geometry (thickness and surface dimensions) of the specimen and the thermomechanical conditions of transformation affect the calibration require further investigation if the technique is to be used where a range of compositions or specimen dimensions might be encountered. Specifically, the effect of the composition of the specimen, its area, thickness, surface finish (roughness), curvature (if any), and possibly the temperature and stress system under which transformation occurred, require investigation.

There was close correlation between the amount of transformation as measured by the balance and the effective strain for tensile specimens oriented at 0, 45 and 90° to the rolling direction (Fig. 3) and for a wide range of strain ratios (Fig. 4), which suggests that the calibration is unlikely to be affected by the occurrence of preferred orientation, or by the stress system under which the steel deformed.

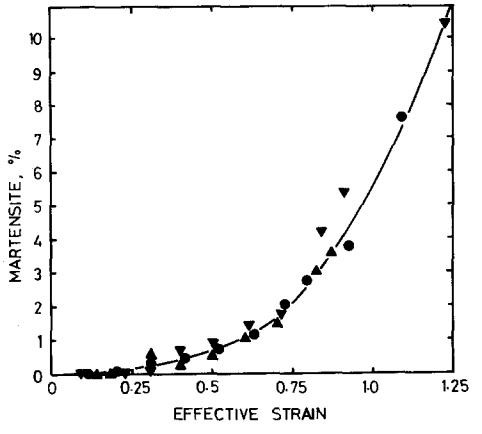

Figure 3 The relationship between effective strain and transformation to martensite in specimens of Type 301 stainless steel tested with the tensile axis at 0° •, 45° ▲ and 90° ▼ to the rolling direction, showing insensitivity to specimen orientation.

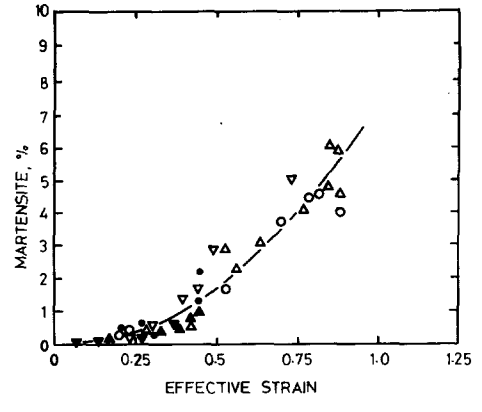

Figure 4 The relationship between effective strain and transformation to martensite in specimens of Type 301 stainless steel, hydrostatically bulged at the maximum elongation temperature for biaxial stress systems (62C), showing insensitivity to strain. Plots for different values of the ratio of in-plane principal strains e_1/e_2 as follows; $e_1/e_2 = 0.875$ ▼, 1.00 ▲, 1.50 ○, 2.00 △, 3.00 ▽, and 4.00 •.

3. Conclusions

As shown by previous workers, use of the three lowest-order reflections of each phase led to a consistent determination of the amount of strain-induced martensite in an 18/8 austenitic stainless steel and the apparent overestimate, resulting from use of only the reflections of lowest order in the austenitic and martensitic phases, agreed with previous work, suggesting that use of the first three reflections in each phase was sufficient to include the significant components of texture.

The difficulty experienced by previous workers in using the techniques of quantitative metallography to determine the amount of martensite present in metastable austenitic stainless steel was confirmed, but measurement of the force required to break contact with a hemispherical probe incorporating a permanent magnet was found to provide a rapid, simple, reproducible and widely-applicable method of determining the amount of α' martensite in metastable austenitic stainless steel.

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