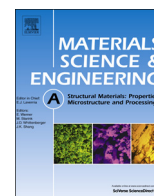




ELSEVIER

Contents lists available at ScienceDirect

## Materials Science &amp; Engineering A

journal homepage: [www.elsevier.com/locate/msea](http://www.elsevier.com/locate/msea)

# Determination of low levels of retained austenite in low-carbon high-manganese steel using X-ray diffraction

Helder Carvalho Ferreira<sup>a</sup>, Francisco Jose Martins Boratto<sup>b</sup>, Vicente Tadeu Lopes Buono<sup>c,\*</sup>

<sup>a</sup> ArcelorMittal Monlevade, Av. Getúlio Vargas, 100, 35930-000 João Monlevade, MG, Brazil

<sup>b</sup> Independent Consultant, Av. Aeroporto, 9, 35930-438 João Monlevade, MG, Brazil

<sup>c</sup> Universidade Federal de Minas Gerais, Av. Antonio Carlos, 6627, 31270-901 Belo Horizonte, MG, Brazil

## ARTICLE INFO

## Article history:

Received 19 October 2014

Received in revised form

8 January 2015

Accepted 9 January 2015

Available online 23 January 2015

## Keywords:

X-ray diffraction

Retained austenite

Pearson VII function

## ABSTRACT

A method involving the decomposition of the X-ray diffraction (XRD) peaks for the single wavelengths  $K\alpha_1$  and  $K\alpha_2$  was used to quantify the amount of retained austenite at levels lower than 5% in low-carbon high-manganese steels. By applying this method, it was possible to use the two main peaks of austenite ( $\gamma$ ) and the two main peaks of ferrite ( $\alpha$ ) in the calculations, despite the partial overlapping of the  $(111)\gamma$  and  $(110)\alpha$  peaks. The diffraction peaks were modeled with the Pearson VII equation using a nonlinear least-squares optimization technique. This allowed the integrated intensities of the XRD peaks to be calculated using only the  $K\alpha_1$  side. The method was used to measure the levels of retained austenite in samples of a metal-inert gas steel welding rod cooled at the rates of 10 °C/s and 1.6 °C/s. The accuracy of the method was determined by performing six measurements in different directions in both the longitudinal and the transverse section of the 1.6 °C/s sample.

© 2015 Elsevier B.V. All rights reserved.

## 1. Introduction

Steels for metal-inert gas (MIG) welding electrodes are usually produced by adding manganese and silicon to a low-carbon steel base and may contain retained austenite ( $\gamma_R$ ), which is associated with martensite, in their as-rolled microstructure. This martensite-retained austenite phase mixture is called the MA constituent, and its presence may increase the hardness of the steel rod, causing it to fracture during wire drawing. This phase can also increase the final strength of the wire to values greater than those desired. Previous measurements in the plant indicate that the best results – direct drawing from 5.5 mm in diameter wire rod to 0.80 mm final diameter – are obtained when the reduction of area in tensile tests of the wire rod is greater than 74.5% and the strength is lower than 573 MPa. For strengths higher than this value, the volume fraction of the hard phase is such that the rod will not sustain deformation until the final size is reached.

\* Correspondence to: Department of Metallurgical and Materials Engineering, Universidade Federal de Minas Gerais, Av. Antonio Carlos, 6627 – Campus Pampulha, 31270-901 Belo Horizonte, MG, Brazil. Tel.: +55 31 3409 1859; fax: +55 31 3409 1815.

E-mail addresses: [helder.ferreira@arcelormittal.com.br](mailto:helder.ferreira@arcelormittal.com.br) (H. Carvalho Ferreira), [francisco\\_boratto@yahoo.com.br](mailto:francisco_boratto@yahoo.com.br) (F. Jose Martins Boratto), [vbuono@demet.ufmg.br](mailto:vbuono@demet.ufmg.br) (V. Tadeu Lopes Buono).

<http://dx.doi.org/10.1016/j.msea.2015.01.019>

0921-5093/© 2015 Elsevier B.V. All rights reserved.

The conventional methods for quantifying the amount of retained austenite in steels can present difficulties when it comes to detecting and quantifying low austenite levels, and a critical comparison of the commonly used techniques such as light microscopy, X-ray diffraction (XRD) analysis, and magnetic saturation measurement has not yet been performed for this type of material [1].

In this study, the classical approach of decomposing XRD peaks modeled with the Pearson VII equation [2–4] while using nonlinear least-squares optimization routines was employed to evaluate the low levels of retained austenite in a MIG electrode steel. Using this technique, experimental XRD peak intensity measurements were performed only on the  $K\alpha_1$  side of the peaks, and Pearson VII function algebraic integration was employed. This made it possible to use the four high-intensity peaks, despite the  $(111)\gamma$  and  $(110)\alpha$  peaks exhibiting partial overlapping. Using a single wavelength,  $K\alpha_1$ , instead of the “weighted averages” of  $K\alpha_1$  and  $K\alpha_2$ , as recommended in ASTM E975 [5], avoids interference of  $K\alpha_2$  in the peak intensity. This interference must be avoided for accurate measurements because it varies with the Bragg angle and is stronger for low index planes. The proposed method was then used to measure the  $\gamma_R$  levels in rod samples of a MIG material cooled at rates of 10 °C/s and 1.6 °C/s. The accuracy of the method was confirmed by performing six measurements at different directions in both longitudinal and transverse sections of the 1.6 °C/s sample.

## 2. Materials and methods

### 2.1. Sample collection and preparation

Samples of the MIG material were collected at the intermediate profile (30 mm and 17.5 mm) from a wire rod rolling mill. Table 1 shows the chemical composition of the alloy, which corresponds to a ER70S-6 grade. Other elements are at residual levels.

Initially, the rod with a diameter of 30 mm was homogenized at 1200 °C for 7 days, in order to eliminate the banded structure. After being cooled to room temperature, it was machined to the standard dimensions used for Jominy tests [6]; this also removed the decarburization layer formed during the heat treatment. A first sample was obtained by austenitization in a laboratory electric furnace at a temperature of 910 °C for 30 min and subsequently cooling in a Jominy test apparatus. A cross-sectional sample was taken at a distance of 18 mm from the cooling end; according to the equation developed by Brian [7], this position along the Jominy bar corresponds to a cooling rate of approximately 10 °C/s at 700 °C. The sample cooled under these conditions was used to develop the proposed XRD method.

Other samples were obtained using the 17.5 mm rod, which was not subjected to the homogenization treatment, but austenitized at 910 °C for 30 min and subsequent cooled in air to room temperature. This simulated the low-end cooling rate of a wire-rod-cooling conveyor. A chromel–alumel thermocouple was inserted in the sample to monitor the temperature. A cooling rate of 1.6 °C/s at 700 °C could be achieved in this manner. This rod was cut in longitudinal and transverse sections.

All the samples were prepared using the conventional procedures, including cutting, grinding, and polishing, and care was taken to avoid heating their surfaces. The XRD analyses were performed on the sample cooled at 10 °C/s (i.e., the sample corresponding to the transverse section of the original rod), as well as on longitudinal and transverse sections of the 1.6 °C/s rod. The measurements were made at the center of the samples, illuminating the same area of 10 mm × 10 mm. For each sample, the data were collected in six symmetrically spaced positions, which were set by rotating the sample around the normal to its surface. This was done to avoid the possible influence of crystallographic texture.

For the measurements, a Shimadzu XRD6000 diffractometer with a Co-K $\alpha$  radiation source was used; diffraction by monochromator and sample take place in the same plane. The scan rate was 0.02°2 $\theta$ /s, each step was 3 s, and scans were made for 2 $\theta$  values of 48–80°. Color metallography was also performed, using a previously described method [8].

### 2.2. Calculation of the austenite volume fraction

If a phase does not have a preferred crystallographic orientation, its XRD intensity is proportional to its volume fraction [9,10] and is given by the following equation:

$$I_{\gamma}^{(hkl)} = K_i R_{\gamma}^{(hkl)} \frac{V_{\gamma}}{2\mu} \quad (1)$$

where:

$I_{\gamma}^{(hkl)}$ : the measured integrated intensity per angular diffraction (hkl) peak in the  $\gamma$ -phase;  
 $R_{\gamma}^{(hkl)}$ : the theoretical intensity of the  $\gamma$ -phase relative to that (hkl) peak;

$K_i$ : a constant and related to the group geometric factors of the instrument and the type of radiation used, but independent of the type of the sample;

$V_{\gamma}$ : the volumetric fraction of the  $\gamma$ -phase;

$\mu$ : the linear absorption coefficient of the sample

**Table 1**

Chemical composition (in wt%) of the welding electrode alloy investigated.

| Classification       | %C        | %Mn       | %Si       |
|----------------------|-----------|-----------|-----------|
| Alloy                | 0.095     | 1.637     | 1.096     |
| ER70S-6 <sup>a</sup> | 0.06–0.15 | 1.40–1.85 | 0.80–1.15 |

<sup>a</sup> European Specification – EN440; American Specification – AWS A 5.18

For steels containing only  $\alpha$  (ferrite/martensite) and  $\gamma$  (austenite) phases, it is possible to calculate the volumetric fraction ( $V_{\gamma}$ ) of the  $\gamma$ -phase from the following equation:

$$V_{\gamma} = \frac{\sum I_{\gamma}^{(hkl)} / R_{\gamma}^{(hkl)}}{\left( \sum I_{\alpha}^{(hkl)} / R_{\alpha}^{(hkl)} + \sum I_{\gamma}^{(hkl)} / R_{\gamma}^{(hkl)} \right)} \quad (2)$$

### 2.3. XRD peaks of interest

Owing to the low concentration of austenite in the studied alloy, only four peaks with high intensities were selected. These were two ferritic peaks, (110) $_{\alpha}$  and (200) $_{\alpha}$ , and two austenitic peaks, (111) $_{\gamma}$  and (200) $_{\gamma}$ . Each peak was decomposed for wavelengths of Co-K $\alpha_1$  and Co-K $\alpha_2$ . Further, in order to increase the accuracy of the calculation of the volumetric fraction of austenite, only the peaks related to the Co-K $\alpha_1$  wavelength were used.

### 2.4. Modeling a diffraction spectrum with austenite and ferrite peaks

To model the peak intensities obtained by the XRD measurements, several equations have been proposed: Gauss, Lorentz, Pearson VII, Voigt, and pseudo-Voigt [11]. Among these, the Pearson VII and pseudo-Voigt equations yield the best results when used for modeling diffraction peaks. In this study, the Pearson VII function was used in the form of the following equation:

$$I(x) = I_0 \left[ 1 + \frac{K^2(x-x_0)^2}{M} \right]^{-M} \quad (3)$$

where  $x$  is the 2 $\theta$  value and  $I_0$  is the maximum intensity of the peak at  $x_0$ . The parameter  $K$  controls the width of the curve, and  $M$  is the decay ratio of the “tails”. For  $M$  values close to one, the curve is purely a Cauchy profile. For  $M$  values close to two, it is a Lorentzian, and for  $M$  values approaching infinity, the profile is a Gaussian one [12]. The peak position  $\theta_{K\alpha_2}$  may be obtained as a function of  $\theta_{K\alpha_1}$  by using the Bragg equation as follows:

$$\theta_{K\alpha_2} = \sin^{-1} \left( \left( \frac{\lambda_{K\alpha_2}}{\lambda_{K\alpha_1}} \right) \sin(\theta_{K\alpha_1}) \right) \quad (4)$$

Therefore, the complete model for  $I(x)$  must include the K $\alpha_1$  and K $\alpha_2$  doublets for both the ferrite and the austenite phases, plus a linear background, ( $a + bx$ ). The final shape for this model, which includes four peaks, is as follows (the odd index refers to the K $\alpha_1$  peak and the even index to the K $\alpha_2$  peak):

$$I(x) = I_{(111)\gamma}(x) + I_{(110)\alpha}(x) + I_{(200)\gamma}(x) + I_{(200)\alpha}(x) + (a + bx) \quad (5)$$

where:

$$I_{(111)\gamma}(x) = I_1 \left[ 1 + \frac{K_1^2(x-x_{01})^2}{M_1} \right]^{-M_1} + I_2 \left[ 1 + \frac{K_2^2(x-x_{02})^2}{M_2} \right]^{-M_2}$$

$$I_{(110)\alpha}(x) = I_3 \left[ 1 + \frac{K_3^2(x-x_{03})^2}{M_3} \right]^{-M_3} + I_4 \left[ 1 + \frac{K_4^2(x-x_{04})^2}{M_4} \right]^{-M_4}$$

$$I_{(200)\gamma}(x) = I_5 \left[ 1 + \frac{K_5^2(x-x_{05})^2}{M_5} \right]^{-M_5} + I_6 \left[ 1 + \frac{K_6^2(x-x_{06})^2}{M_6} \right]^{-M_6}$$

Download English Version:

<https://daneshyari.com/en/article/7978304>

Download Persian Version:

<https://daneshyari.com/article/7978304>

[Daneshyari.com](https://daneshyari.com)