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Austenitemeter – Mössbauer spectrometer for rapid determination of residual austenite in steels

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ABSTRACT

In this note we describe an application of backscattering ^{57}Fe Mössbauer spectroscopy for determination of the austenite content in steels, where the surface analysed thickness is ~20 μm. The method enables rapid determination of the residual austenite in the sample, i.e. within 10–20 min with a sufficient accuracy. Calculations using different *f*-factors for austenite and ferrites are also adopted. Standard regime of the analysis allows determining an austenite-to-ferrite ratio with a high precision after several hours or few days of measurement for laboratory research or quality control purposes. The method demonstrates high efficiency and more sensitive results of the analysis, i.e. compared with the XRD method. Statistical quality of the spectra which affects the measurement accuracy (±1%) with detection limit of rapid method (2–3% austenite) together with noise occurrence is evaluated.

1. Introduction

Backscattering ^{57}Fe Mössbauer spectroscopy (MS) allows non-destructive [1,2] material analysis for study of both large and small samples, for determination of iron-bearing phases occurrence with the accuracy below ±1%. MS applications in metallurgy and engineering are really significant for determination of residual austenite content [3]. In such types of analysis (performed with ^{57}Co radiation source) mainly X-rays 6.3 keV are registered, where their penetration is about 1–20 μm in the sample [4]. In addition, depth selectivity can be obtained by utilizing another appropriate type of scattered radiation, such as re-emitted gamma-rays 14.4 keV (~100 μm and internal conversion electrons 7.3 keV (~300 nm) [2,5]. It is common knowledge that utilization of conversion electrons is basically destructive for the sample as, according to this method, a sample should be placed into the body of a detector.

Austenite, a gamma form of iron, occurs mostly in the form of soft and ductile grains. It is present in the most commonly used types of stainless steel. Even small content of austenite (residual austenite) has an impact on strength and ductility of the steel. Residual austenite is a by-product of martensitic transformation, where small portion remains unconverted. The following heat treatment – tempering is performed in order to convert the residual austenite to bainitic type structure. The disadvantage of this heat treatment is the increase in the specific volume of the grains of austenite and the release of heat. On the contrary, the internal stress decreases. Depending on the amount of residual austenite, the hardness of the steel can be reduced or increased.

The study of stabilization processes of retained austenite in steels [6,7] is important for long term properties of steel used in constructions and engineering devices. In order to enhance the ductility of high-strength steels it was shown that it is necessary to increase their content of retained austenite. For instance, alloys with high ductility and excellent levels of mechanical strength can be obtained by the transformation of austenite to martensite during plastic deformation [8]. Hence transformations of retained austenite are studied to promote better mechanical properties of steels. Materials can also undergo phase transformations during machining operations with cutting tools, and thus the retained austenite can occur [9]. Presence of retained austenite as the additional phase in dual phase steels (customarily a ferrite–martensite mixture) has been thought to be beneficial as it improves toughness of the steel by taking recourse to transformation induced plasticity phenomenon [10].

In the field of steel characterization, the Mössbauer spectra are mostly evaluated as a superposition of paramagnetic single line associated with austenite and complex magnetic split pattern with broad asymmetric lines assigned for ferrites [11–13]. The magnetically split subspectra belong to Fe atoms, which have different neighbourhood of alloying elements [14,15]. The MS is able to quantify the relative amount of the austenite and ferrite phases

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Table 1
Heat treatment procedures of the samples (quenched to 85 °C in oil for 5 h).

Sample	Austempering	1. Tempering	2. Tempering	3. Tempering
A	930 °C/10 h	–	–	–
B	930 °C/10 h	530 °C/14 h	–	–
C	930 °C/10 h	530 °C/14 h	540 °C/10 h	–
D	930 °C/10 h	530 °C/14 h	540 °C/10 h	510 °C/10 h

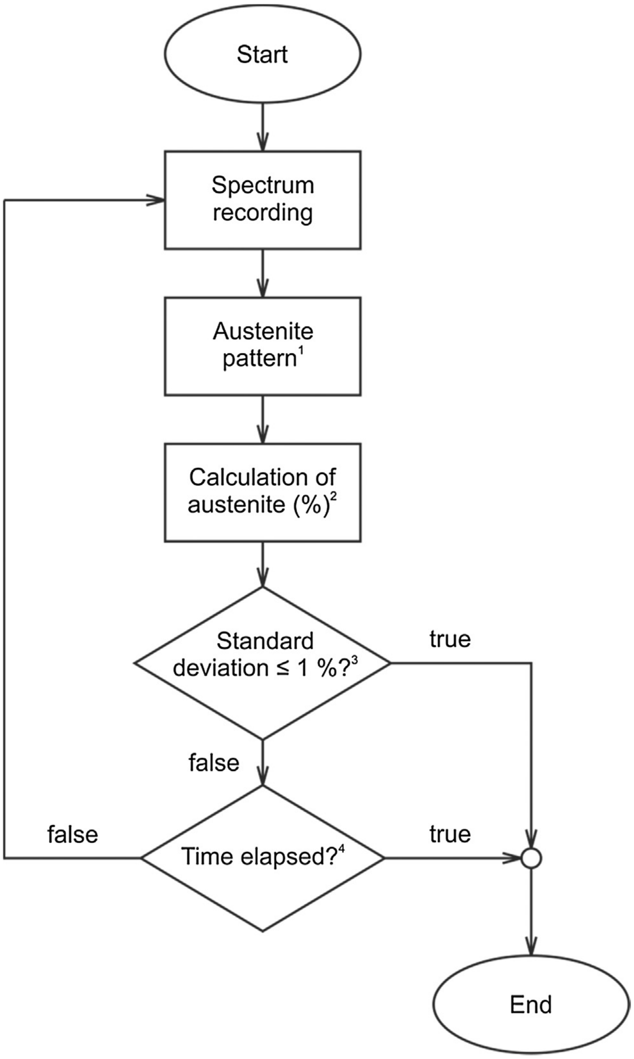

Fig. 1. The flow chart of the characterization procedure for rapid determination of residual austenite in the on line analysis of Mössbauer spectra.

on the basis of their totally different magnetic properties in a way, which is relatively independent of the shape, size, and orientation of the particles [2,16]. To obtain the relative phase fractions by the conversion of spectral area fractions, corrections for recoil-free fractions and phase compositions are also required. Fortunately, those effects are expected to be small, in the order of a few percent, and thus can be neglected for practical purposes [2].

Many so far studies were focused on the recoilless fractions of iron-phases in steels [2,10,17] and are not consistent. The calculation of the real iron phase compositions for austenite and ferrite can be adopted from [10,17], where the relative quantity of the γ-phase (austenite) M_γ was determined by measuring the relative areas of the subspectra according to the following equation:

$$M_\gamma = 100\frac{A_\gamma}{A_\gamma + dA_\alpha}, \tag{1}$$

where A_γ and A_α are the areas of γ- and α- (ferrite) phases, while $d = 0.86$ takes into account the difference between the Debye-Waller factors of those phases [2,10,17].

The development of rapid, on-line (meaning without fitting techniques and off-line analysis by extra software), and reproducible measurement technique of the absolute austenite content is a challenge. In the context of the historical terminology where similar unique instruments named as ferritometer [16] or pyritemeter [18] were designed, we can name this developed system a Mössbauer austenitemeter. Such instruments are based on the precise knowledge of iron-phases hyperfine parameters expected in the sample. After identification of such phase (austenite, ferrite, martensite, pyrite, etc.) its relative content to other phases in the sample is calculated. This calculation is based on the optimized electro-mechanical construction of the spectrometer [16,18] or on the optimized numerical processing of the resulted spectra [19].

Quantitative X-ray diffraction analysis is also often employed to determine the relative amount of residual austenite and occurrence of ferritic phases, but the Mössbauer results for the austenite content can differ significantly from XRD results due to a crystallographic texture effect that has a strong impact on the XRD measurements [8]. The sensitivity and accuracy of the determination of austenite by MS is especially high in the case of small austenite grains [14]. Nevertheless, we believe that a combination of methods used to determine and verify the true austenite content in steels provides the best results.

As a standard industrial method, XRD allows to analyse 10–15 specimens per day since each takes from 30 to 60 min on average. However, only small specimens or powdered samples can be measured. Quality of the results is user independent with inaccuracy even below 0.5%, however it requires an expert operator in XRD software application and data analysis. In industry, the austenite content is preferably determined by metallographic methods employing an optical microscope. The metallographic methods then also allow the grain size analysis. However, disadvantages of this method can lie in the facts that only a few specimens per day can be analysed, and it is destructive since the samples have to be prepared by both mechanical and chemical processing. Quality of the results depends on the usefs experience and the method inaccuracy is 10–15%.

This note is focused on the investigation of the applicability of the Mössbauer backscattering spectroscopy method for determination of the content of austenite in ferrite.

2. Experimental and methods

Four samples named as A, B, C, D (2 cm high, 7 cm long, 3 cm wide) of a 50Cr5MoV steel were prepared and thermally treated in an external laboratory, following the processes presented in Table 1. No further mechanical and/or chemical processing was employed.

The iron-phase analysis of the samples was established by means of Mössbauer spectrometer [20] continuously developed for industrial use and newly developed toroidal gas-flow X-ray counter [21] with enhanced counting efficiency of 6.3 keV X-rays re-emitted from the irradiated sample. Measurements were per-