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A Magnetic Force Microscopy Study of Magnetic Domain Structure in Maraging Steel

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Abstract. This study presents the results of an analysis of the domain structure of supersaturated X2CoCrMoAl20-15-3 maraging steel. The analysis was carried out using the magnetic force microscopy method in a two pass mode (surface topography and subsequent magnetic phase shift). Obtained results were subjected to numerical processing. The size of spontaneously magnetized regions and their orientation were determined by analyzing the autocorrelation function of the magnetic phase shift of the oscillating tip. The structure of magnetic domains was then described using fractal parameters such as fractal dimension and topothesy.

Introduction

Maraging steels are an attractive structural material with a large number of applications in the arms industry, aviation and astronautics. They are characterized by excellent mechanical properties, including Young's modulus of up to 2 GPa, hardness of up to 60 HRC and high ductility. The above properties are shaped by precipitate strengthening during which supersaturated martensitic structures are reinforced with intermetallic compounds in the aging process.

This group of structural materials includes among others X2CoCrMoAl20-15-3 steel which is characterized by very good mechanical properties (Rm = 1.5 GPa, 48 HRC) and resistance to corrosion. This type of steel, also known as Pyromet X-15, was researched intensively in the 1960s and 1970s, but is rarely used today, mainly due to the high price of cobalt and low fracture toughness. However, its unique structural properties were discovered only recently with the advance of modern analytical methods, such as scanning probe microscopy, which might open new perspectives of its use.

Materials and Methods

The experimental material consisted of a supersaturated X2CoCrMoAl20-15-3 alloy. Samples were supersaturated at the temperature of 1050°C for 30 minutes and cooled to ambient temperature in water. Polished samples were analyzed by an AFM (Atomic Force Microscope) Multimode 8 microscope (Bruker) in the MFM (Magnetic Force Microscopy) mode. The measurements were performed using a two pass method: during the first pass, the surface topography was scanned in a tapping mode, and during the second pass (magnetic force imaging), the probe was lifted 100 nm above the surface to eliminate Van der Waals forces. When a oscillating probe is moving above a magnetized region, a phase shift in its oscillation frequency occurs [1], which is described by Eq. 1:

$$\Delta\Phi = \frac{Q}{k}\left(q\frac{\partial H_z}{\partial_z} + m_x\frac{\partial^2 H_x}{\partial_z{}^2} + m_y\frac{\partial^2 H_y}{\partial_z{}^2} + m_z\frac{\partial^2 H_z}{\partial_z{}^2} \right) \tag{1}$$

where: $\Delta\Phi$ – measured phase shift, Q – quality factor of an MFM probe, k – spring constant, q – effective magnetization of probe, m_x, m_y, m_z – magnetic moment of an MFM probe, H_x, H_y, H_z – components of magnetic field above the sample.

The recorded phase shifts support mapping of spontaneously magnetized regions on the surface of the studied material [2]. Six randomly selected regions with side length of 2, 5, 10, 20 and 50 μm were scanned. The width of magnetic stripe domains was determined by analyzing the spectra of autocorrelation function R(k,l) of the magnetic phase shift signal defined as:

$$R(k,l) = \frac{1}{MN}\sum_{x=0}^{M-1}\sum_{y=0}^{N-1}\left[z(x,y)-z_m\right]\left[z(x+k,y+1)-z_m\right] \tag{2}$$

where: M and N – number of samples in scan directions; x and y – discreet coordinates of a given point in the image; z(x, y) – phase shift at a given point in the image; z_m – average phase shift. The spectral width of magnetic domains was specified as full width at half-maximum (FWHM) of function R(k,1) [3].

Results

The MFM analysis revealed fluctuations in the magnetic field emitted by spontaneously magnetized regions of the steel sample 100 nm above its surface (Fig. 1). Magnetic domains with the length of 1÷4 μm were observed. Orientation of magnetized domains gave rise to magnetic anisotropy of the sample. The degree of anisotropy was determined based on autocorrelation function R(k,l) in terms of the anisotropy ratio S_{tr}, described by Eq. 3. The anisotropy ratio is expressed as a proportion between two decay lengths τ_{min} and τ_{max} measured along a_1, and a_2 axes, respectively, at which the value of R(k,1) falls down from 1.0 to 0.2 [4]:

$$0 < S_{tr} = \left.\frac{\tau_{\min}}{\tau_{\max}}\right|_{R=1\to 0,2} \le 1 \tag{3}$$

The spectrum of R(k,l) plotted for the images with scan size L = 5, 10, 20 and 50 μm exhibits nearly equiaxial shape, which implies isotropic magnetic domains. For smaller scan size (L = 2 μm), the spectrum is clearly asymmetrical, suggesting high anisotropy at that level. The spectrum of R(k,l) is shown in Fig. 2 together with a method for measuring the domain width (FWHM).

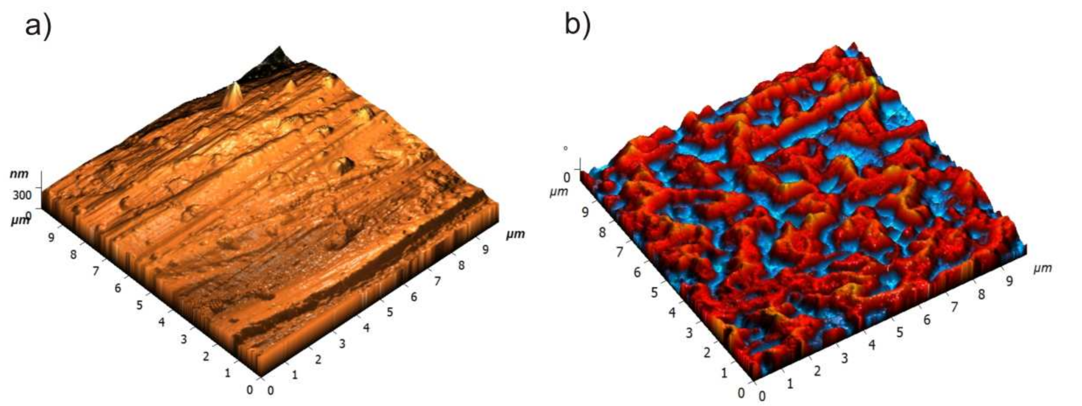

Fig.1. MFM results of X2CoCrMoAl20-15-3 steel, a) surface topography; b) magnetic domains structure.

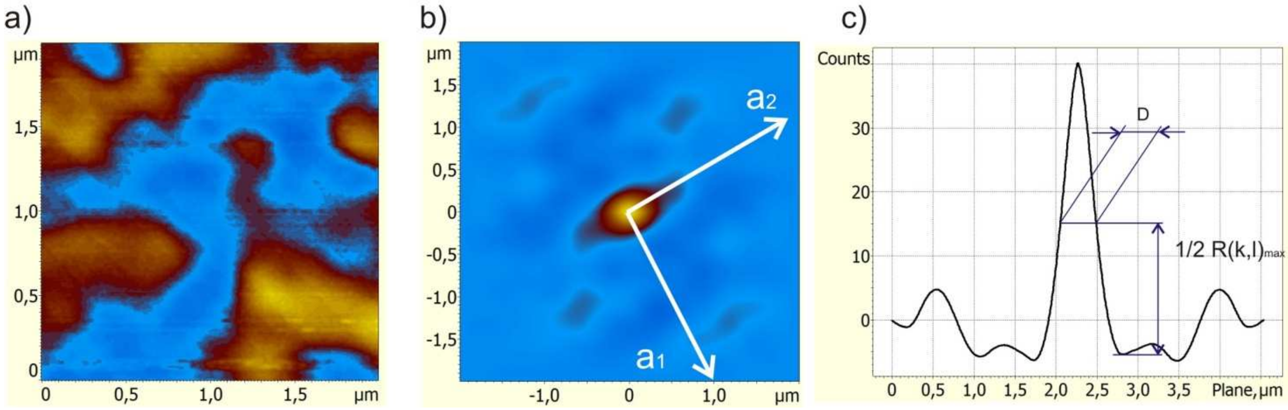

Fig.2. Domain structure of X2CoCrMoAl20-15-3 steel: a) autocorrelation function b) method for measuring the FWHM of magnetic domains.

The changes in the domain anisotropy are presented in Fig.3. In regions larger than 5 μm, the anisotropy ratio S_{tr} moves asymptotically towards 0.833 and does not scale with the scan size, therefore, it could be used as a parameter describing the domain structure. Its value indicates that observed magnetic properties are rather isotropic. Similar dependence between S_{tr} vs. L was reported by Bramowicz in a morphological analysis of a martensitic structure [5]. Nonetheless, further work is needed to describe changes in S_{tr} in the transition range from 2 to 5 μm. In our study, the average width of magnetic domains in the main directions of anisotropy reached 440 and 523 nm in the direction of the slowest (a_1) and the fastest (a_2) decay in the autocorrelation function, respectively. A fractal approach is therefore suggested to describe observed domain structure using 2-dimensional structure function given by Eq. 4 [4]:

$$S(k,l) = \frac{1}{MN}\sum_{m=0}^{M-1}\sum_{n=0}^{N-1}\left[z(m,n) - z(m+k,n+l)\right]^2 \tag{4}$$

To determine fractal properties such as: fractal dimension *F* and topothesis *K*, 1-dimensional profile structure functions *S(τ)* are needed, calculated by averaging all possible intersections of S(k,l) along a given direction. Profile structure function can be expressed by fractal parameters in the following form [4]:

$$S(\tau) \propto K^{2(F-1)}\tau^{2(2-F)} \tag{5}$$

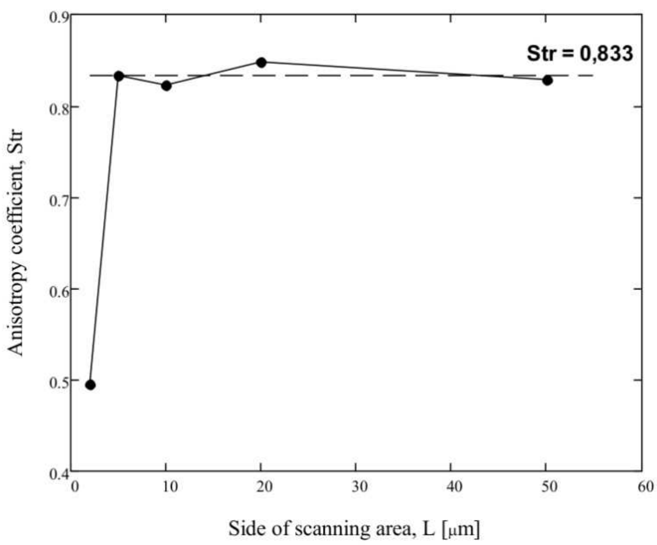

Fig.3. Changes in the anisotropy ratio of magnetic structure as a function of scan size.

Structure functions mapped along main directions of anisotropy with the values of fractal parameters determined in a series of magnetic measurements are presented in Fig. 4.

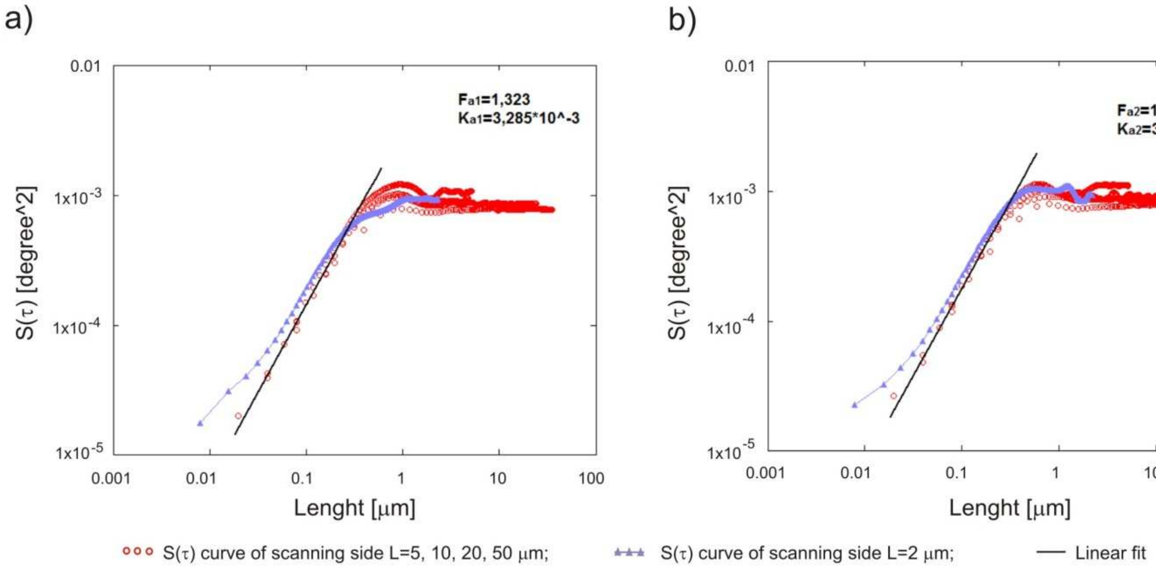

Fig.4. Structure functions of the analyzed domains mapped along, respectively a_1 (left) and a_2 (right) direction.

As demonstrated by Fig. 4, curves S(τ) for scanned regions with sides longer than 5 μm have a typical shape because their linear ranges nearly ideally overlap, which indicates that domains whose sides are longer than 5 μm have fractal character. The structure function of a region with side length L = 2 μm clearly deviates from the typical range. The domain structure of the studied steel sample is, therefore, a multifractal object, and its fractal geometry can be analyzed in the range of 5÷50 μm.

Conclusions

The results of the presented magnetic force microscopy study exhibit domains of supersaturated X2CoCrMoAl20-15-3 steel that are highly isotropic structures comprising stripe domains with the length of 1÷4 μm and the width of 440÷520 nm for scan size larger than 5 μm. However, for scan size smaller than 2 μm the magnetic picture become to a higher degree anisotropic. As a result, anisotropy analysis reveals transition from anisotropic to isotropic domain structure, while fractal analysis suggest that domains are multifractal objects that can be described independent of the scale length. Fractal parameters create new possibilities for characterizing changes in domain structures caused by external factors such as magnetic field, stress or temperature.

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