Quantitative Microstructure Examination of Cr-Ni-Mo Based Alloy Using EBSD Analytical Technique

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High-alloyed Cr-Ni-based stainless-steel cast alloys are frequently used in thermoelectric installations such as conventional and nuclear power plants. Many years of exploitation of mechanical equipment in these objects have shown that the toughness of these alloys decreases with the operating time and temperature. These alloys have a characteristic duplex structure consisting of austenite and =-ferrite[1]. The =-ferrite content depends on the chemical composition of the alloy and on metallurgical factors, namely manufacturing technology and the exploitation conditions. Therefore, in alloys with a chemical composition the allowed ranges of content of alloying elements completely different microstructures can form. The result of this could be different mechanical properties of a material and different behaviour during its exploitation.

The aim of our investigation was to determine the microstructure and to estimate the amount of austenite and =-ferrite. Individual microstructure constituent was quantitatively defined by three different methods and compared. Significant discrepancy between results obtained by each method is commented and explained in this article. Proportion betweenaustenite and =-ferrite was estimated by using empirical formula, further microstructure was investigated using optical microscopy on polished and chemically etched surface and finally Electron Backscatter Diffraction (EBSD) technique was applied and the amount of each phases was determine crystalographically.

The samples of a Cr-Ni-Mo-based cast alloy, CF-8M-type (ASTM A351), were prepared with a standard casting procedure. The samples for mechanical tests were made and on one of such samples the specimen for methallographic examination was taken near the fracture region.

On the basis of well-known empirical correlations (equation (1) and (2)) the Cr and Ni equivalents, as well as the =-ferrite content were calculated[2]. According to chemical composition the portion of =-ferrite was estimated to 28 %.

$$CR = \frac{w(Cr_{eq})}{w(Ni_{eq})} = \frac{[w(Cr)+1.5w(Si)+1.4w(Mo)+w(Nb)-4.99]}{[w(Ni)+30w(C)+0.5w(Mn)+26(w(N)-0.02)+2.77]} \quad \textbf{(1)}$$

$$F = -68.768+157.909CR-133.171CR^2+47.1849CR^3 \quad \textbf{(2)}$$

Figure 1 (a) shows microstructure of examined steel after etching by potassium hydroxide and potassium ferricyanide. Images analyses of etched microstructures on several field of view gives 45 % amount of =-ferrite, however the EBSD phase analyses (see Figure 1 (b)) on polished surface is much more close to the empirical calculation. Using EBSD techniques it is expected that on perfectly prepared surface the result is due to clear separability of BCC and FCC crystal phase structures beyond a doubt 31,5 % of =-ferrite phase. How can the discrepancy between the OM and SEM/EBSD measurements be

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explained? In order to obtain good contrast between austenite and =-ferrite phases in optical microscope micrograph, it is necessary to use very aggressive etchant which attacks only one phase. The step between unattached austenite and etched ferrite is high which may also cause etching to the austenite phase close to grain boundaries and it is consequently coloured brown like the ferrite. Therefore, the portion of ferrite phase is thus enlarged regarding to austenite phase. Figure 2 shows the inverse pole figure map of both phases with two EBSD patterns and orientation legend for cubic crystal structure. The rainbow coloring inside the same grains indicates that the plastic deformation might appear during the tensile test. Misorientation in each grain is of an order of 10 degrees.

Task as simple as the quantification of certain microstructure constituent can sometimes prove to be very tricky. Some previous studies have shown that measuring the amount of retained austenite by EBSD might not be as easy as it looks due to its transformation on the very surface during the sample preparation. However, in our case EBSD gives the most reliable results.

1. M. Vasudevan et al., Journal Materials Processing Technology, 142 (2003), p20.
2. International standard EN ISO 8249:2000E. Further text goes here.

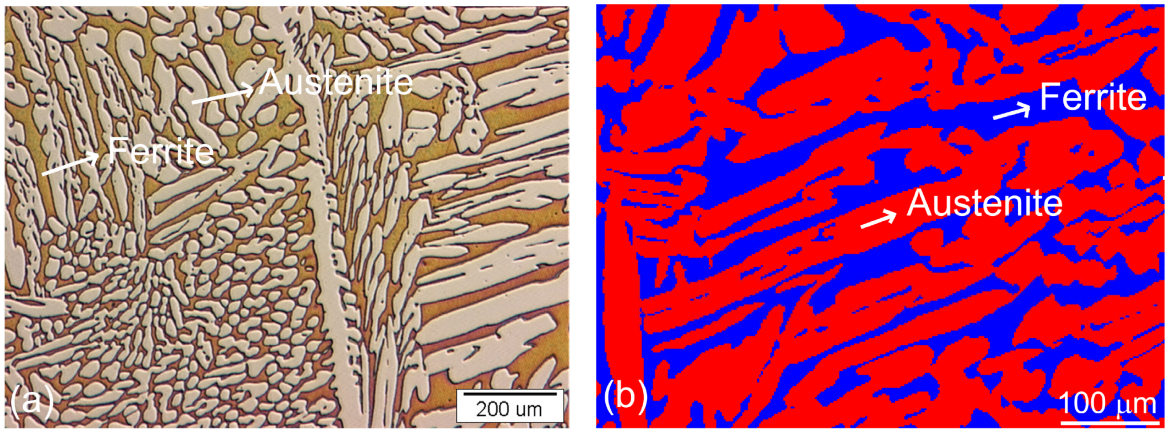

Figure 1. (a) Optical microscopy image of sample microstructure which consist of austenite (white, unattached) and =-ferrite (brown, attached) phases. Etched by KOH 30g, K3(Fe(CN)6) and H2O. **(b)** SEM/EBSD mapping of the same sample (different area) shows the area fraction and the distribution of both phases which were determined by solving the EBSD patterns.

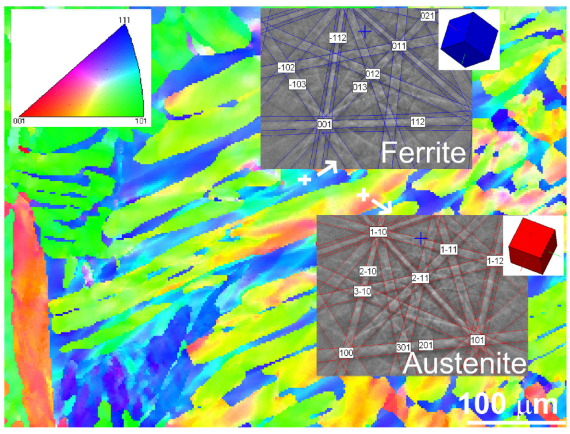

Figure 2. Inverse pole figure map of two phases with EBSD patterns and legend.

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