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GRAIN SIZE ANALYSIS OF Sr-HEXAFERRITES

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ABSTRACT

The quantitative analysis of exaggerated Sr-hexaferrite grains which are randomly oriented in a matrix of small grains is described. The images for analysis were prepared by means of optical microscopy (polarized light) and computer processing. The lengths of the main and perpendicular axes, as well as the aspect ratio of individual grains oriented parallel to the plane of polishing were measured. Due to the much better image contrast obtained and improved ability to identify grain boundaries, the accuracy of measurement is highly increased.

Key words: aspect ratio, image processing, polarization, Sr - hexaferrites

INTRODUCTION

Sr hexaferrites, also well known as hard ferrites, are still one of the most technically important and widely used ceramic materials. Their hard magnetic nature connected to their high magnetocrystalline anisotropy, enables their application where large demagnetizing fields are present (motor segments, flat loudspeakers) (Kools, 1986).

Apart from the intrinsic properties of the magnetic material, microstructural parameters such as grain size and their distribution, shape factor, volume fraction of the ferrite phase, as well as the degree of c-axis alignment of the ferrite grains, influence strongly the magnetic properties (Stablein, 1982). The coercivity strongly depends on grain size and its distribution, while the remanent magnetization depends mostly on the degree of grain alignment and the fraction of the ferrite phase. For optimal magnetic properties an average grain size of about 1 μm and the narrowest grain size distribution has to be attained, together with a high density. In the sintering process a compromise between these requirements has to be achieved. From practical experience as well as literature data (Stuijts, 1956, Kingery, 1976), it is well known that exaggerated grains are very often present in this material. Different authors offered several explanations for the exaggerated grain growth (Stuijts, 1956, Lacour and Paulus, 1975, Kools, 1985, den Broaeder and Franken, 1980, Besenič ar et al., 1989), but no correct definition of this phenomenon was established. It was shown in our previous paper (Besenič ar et al., 1989) that the properties of the starting powder, particularly the average particle size and its distribution, have a remarkable

influence on the microstructural development during the sintering process. For these studies quantitative analysis of the microstructure was of great importance and the present paper deals with a more detailed discussion of such analysis.

Several stereological shape parameters related to nonequiaxial grains have been previously suggested (Underwood, 1970). When the microstructure has a texture, a shape factor can be defined as the ratio of intercept lengths parallel and perpendicular to the orientation axis. However, the analysis of anisometric ceramic microstructures with randomly oriented grains seems to be more complicated, since the standard stereological approach i.e. measuring the intercept length, does not produce exact data.

The present work was performed as part of a study of the effect of powder characteristics on the sintering rate and grain growth, with emphasis on exaggerated grain growth (Besenič ar et al., 1989). Hence, reliable and accurate grain size analysis was of great importance. The aim of the present study was to improve the method of evaluation of the microstructure of Sr-hexaferrite with exaggerated grains.

METHOD

The first problem to be solved before stereological analysis is how to obtain an appropriate image. This requires perfect metallography, since remaining scratches and pull-outs in the microstructure can seriously influence the results. It has to be pointed out that a broad knowledge of the material is necessary to identify the grain boundaries if semi-automatic method is used, although the samples are chemically etched. If an automatic method is utilized, it requires a pretreatment of the grey levels to eliminate the noise and extract the objects to be analysed. Then it is necessary to find a method of segmentation which gives a correct binary image. Thus, although longer and with fewer measurement possibilities, semi-automatic methods are sometimes the only ones usable when the image is too difficult to segment. That is the case with the Sr-hexaferrites.

As can be presumed from Fig. 1 exaggerated Sr- hexaferrite grains in a matrix of small grains grow preferentially in direction of the c - axis. Therefore, the most exact parameter for evaluation of such exaggerated grain growth would be the increase of c-axis length of individual grain and its normal.

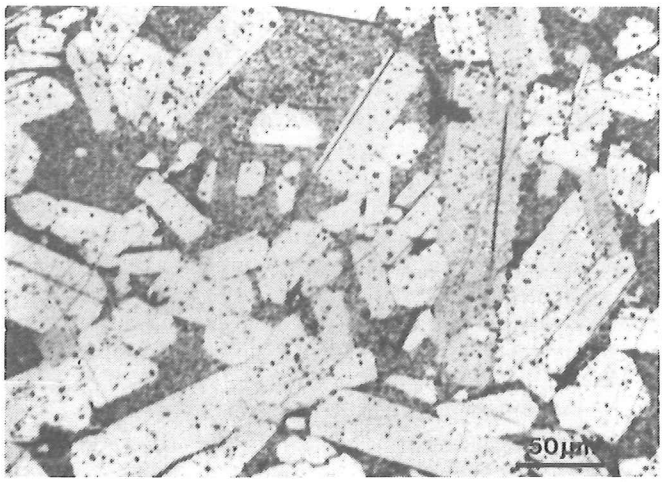

Fig. 1: The etched microstructure of Sr - hexaferrite sintered at 1230°C for 1 hour

Since grains with the profiles of a double hexagonal pyramid are randomly oriented in the structure and cut with the polished plane in various directions, only a few of them are cut parallel to the c - axis. Consequently, stereological analysis of the average intercept length and even the average D_{max} of the grain profile and its normal cannot give the real value of the hexagonal grain length.

Therefore, for present purposes, the grain growth was estimated by evaluation of the aspect ratio of exaggerated grains at different steps of the sintering process, by measuring the c- and a- or b- axis length of individual grains. Regarding the geometry of exaggerated grains (see Fig. 2), it can be proposed that a- (b-) and c- axes correspond to the Ferret diameters D_{min} and D_{max}. The aspect ratio, expressed by D_{min} vs. D_{max} of an individual grain, approaches 1 for equiaxial grains and decreases with the elongation of the grains.

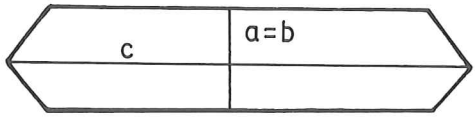

Fig. 2: Schematic presentation of Sr-hexaferrite grain profile

According to the above discussion semi-automatic analysis was used to characterize the Sr-hexaferrite microstructure by means of optical microscopy (polarizing light) using an image analyser IBAS 2 (Opton). To improve the reliability of analysis, computer processing (subtraction) of the obtained images was used.

IMAGE PROCESSING

As one can observe from Fig.1, the optical micrographs of Sr- hexaferrite samples are rather unsuitable for significant image analysis due to low contrast and poorly defined grain boundaries. Semiautomatic analysis could be tiring and of low reproducibility, while on the other hand automatic analysis is not usable for the same reasons.

More suitable micrographs were achieved by using a polarized light microscope. The reflection of Sr hexaferrite grains differs with the varying position of polars, depending on its orientation. To achieve maximum effect, two positions were selected (see Fig. 3a, b). Significant differences in grey tone appeared between individual grains, resulting in a well developed microstructure. Some grain boundaries became visible due to the different grey tone of neighbouring grains. The highest level of contrast was achieved by computer subtraction of two images, resulting on Fig.4. Obviously, grains with the same reflectivity under different angles remain grey, while others for which reflectivity differs, become more contrasty. When compared to Fig. 1, as well as Figs.3 a and 3 b, the computer processed image shows much higher contrast, which enables more accurate measurements. Grain boundaries between the grains with different or the same orientation are clearly visible and identification of grains with their c - axis in the plane of the polished surface is possible. Additionally, the grains which intersect an adjacent grain with different orientation can be detected.

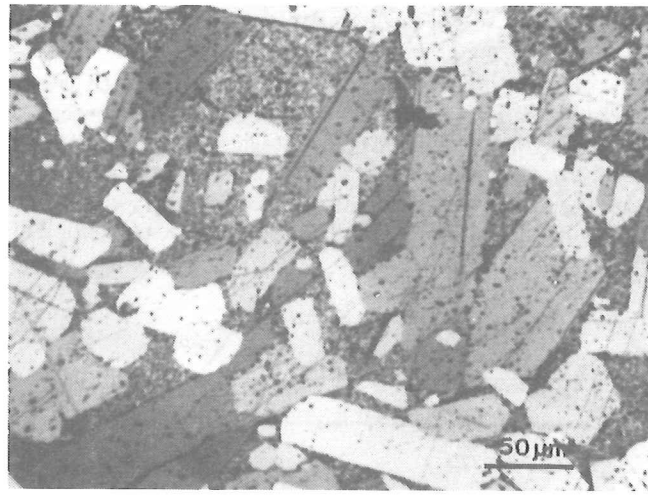

Fig. 3a: The microstructure of Sr - hexaferrite sample (crossed polars for 90^{o})

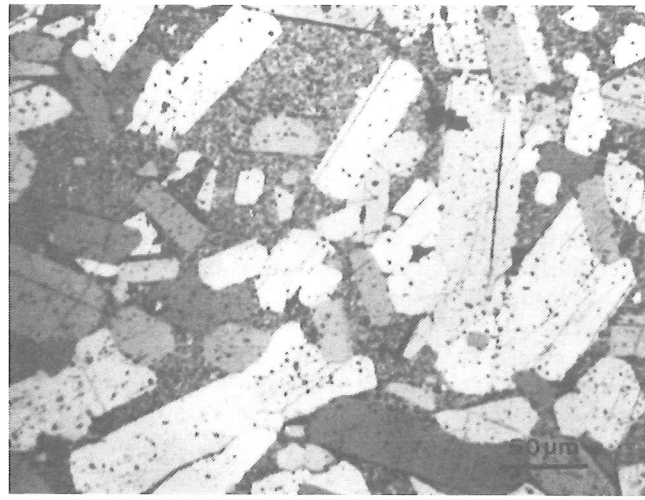

Fig. 3b: The microstructure of Sr - hexaferrite sample (crossed polars, rotation of analyser for 20^{o} regarding to Fig. 3a)

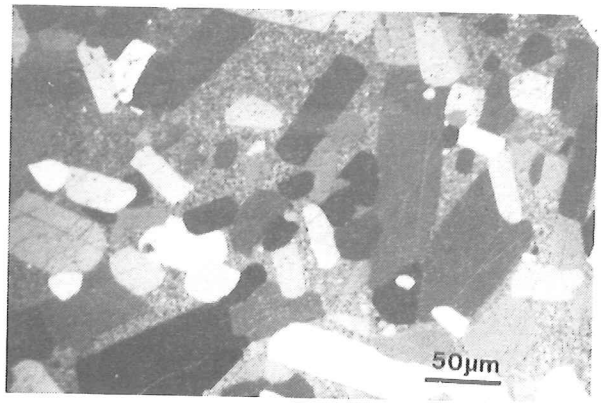

Fig.4: The computer image processed, the subtracted images shown in Fig. 3a and 3b

RESULTS

The characteristic results of semi-automatic grain size analysis of a Sr - hexaferrite sample (sintered at 1230°C for one hour), image processed by the above mentioned method, are shown in Fig.5. Each measurement was repeated on different parts of the sample in view of avoiding errors due to possible homogeneity of the sample. Results presented in Fig. 5 were reproducible. The given parameters were evaluated by outlining the contours of individual large grain exceeding 10 μm in diameter. The parameter D_{min} expresses the approximate length of the a- axis of the particles, while D_{max} should represent the length of the c- axis. The ratio D_{min} vs. D_{max} is a measure of grain growth in one direction.

As shown, a rather wide distribution of aspect ratios was obtained, which could be the consequence of the random orientation of large grains in the matrix, resulting in an apparently shorter c- axis of some grains. In contrast, according to the proposed geometry of hexagonal grains, we suppose that the value of D_{min} approximately agrees with the real a- axis when a computer processed image is evaluated. By measuring grains with the highest contrast, for which it is supposed that they lie in the polished plane, we established that the aspect ratio is approximately 0.4 for almost all measured grains. In addition, considering this value and the D_{min} values of all measured grains in a microstructure, it would be possible to estimate the real c - axis lengths of grains which are oriented out of the polished plane as well. Consequently, the method allows a relatively good description of grain dimensions.

Fig. 6 shows a comparison between the results obtained by semi-automatic evaluation of an etched sample before image processing and after, performed by the same experimenter. A large discrepancy resulting from the much better contrast and from the ability to identify the grain boundaries in the second case is evident. Hence, due to image processing, the reliability and the accuracy of quantitative microstructure analysis is highly increased.

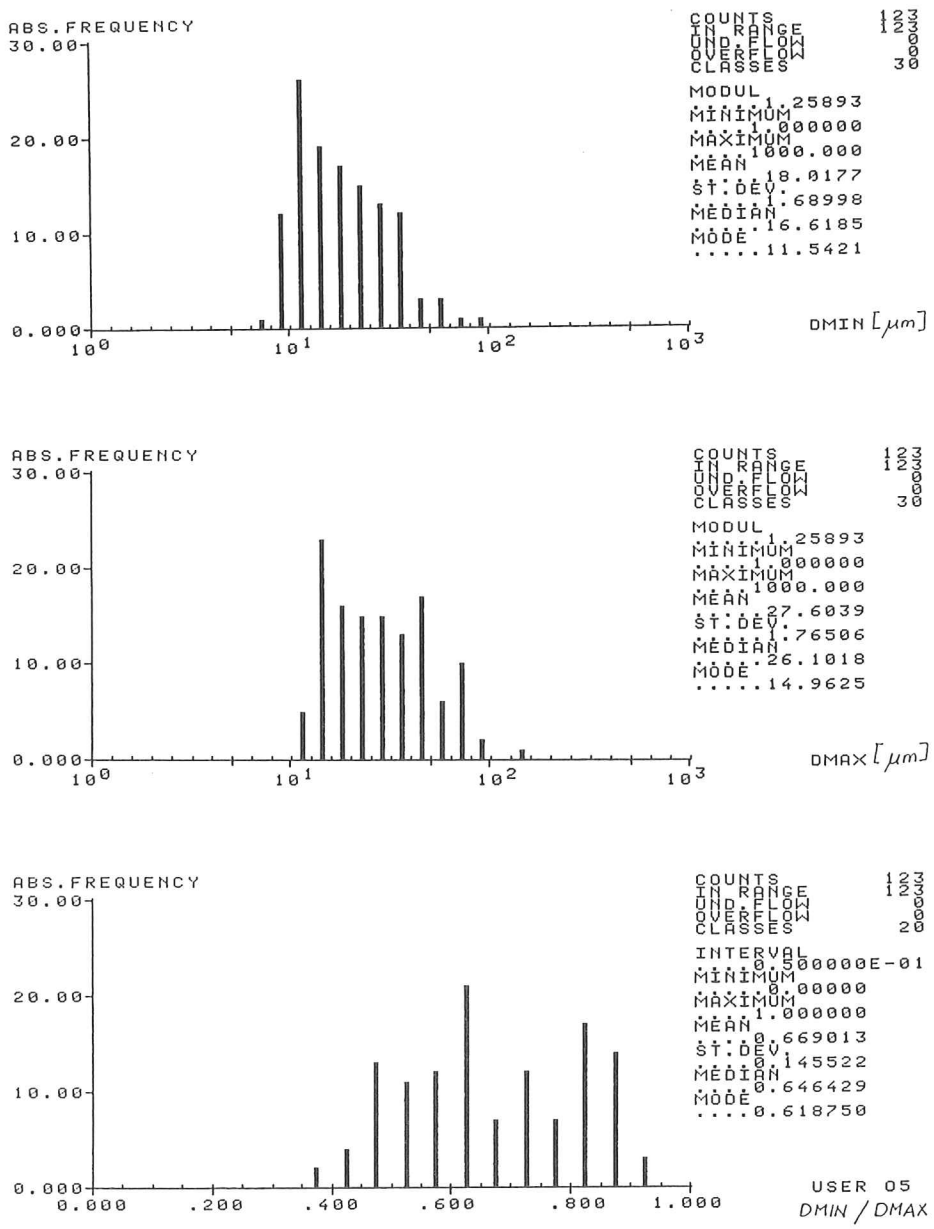

Fig. 5: Characteristic size distributions of computer processed images for Sr hexaferrite sintered at 1230°C, one hour. The diagrams show the distributions for an analysed field with 123 particles. Due to this low number, the distributions are not really statistically representative of the sample.

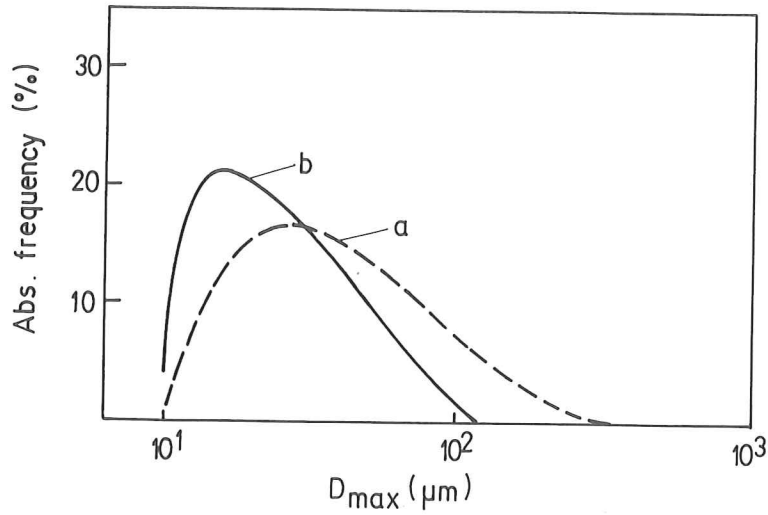

Fig. 6: The comparison of results obtained by the evaluation of images before (a) and after (b) computer processing

The possibility of automatic image evaluation was additionally examined. Fig. 7 shows a computer digitized and processed image of Fig. 4. Obviously, large corrections of a digitized image should be performed before measurement, which would still hardly agree with the real microstructure. Therefore the automatic evaluation of such a microstructure is not recommended even in this case.

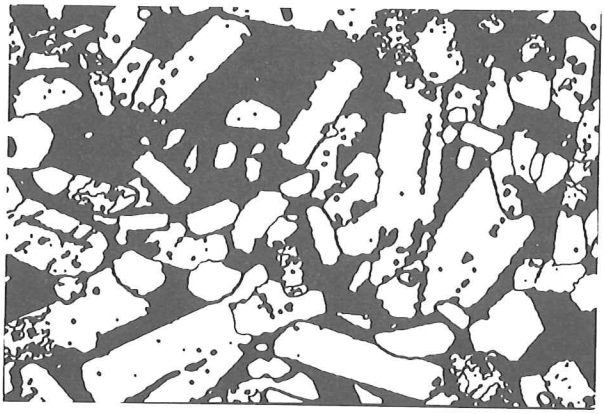

Fig. 7: The digitized image of microstructure shown on Fig. 4

The results of the analysis of exaggerated grain growth of Sr - hexaferrite samples as a function of sintering temperature are shown in Fig.8. As evident, the c - axis of the large grains increases with sintering temperature, while the average aspect ratio remains constant. This corresponds to a constant value for the aspect ratios of selected grains, which it is proposed are oriented in the polished plane. This implies normal grain growth in the investigated temperature region.

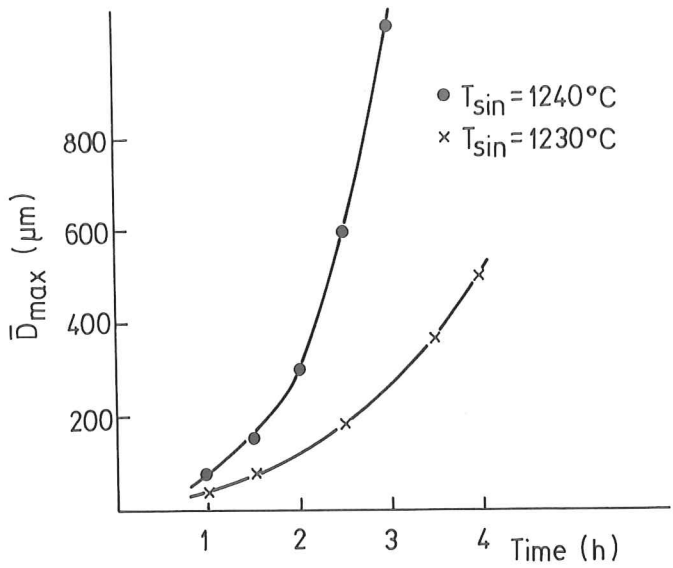

Fig. 8: The increasing of c- axis (expressed by D_{max}) with increasing of sintering temperature

CONCLUSIONS

The semi-automatic analysis of Sr- hexaferrite requires well prepared images to obtain correct results. This can be achieved by computer processing of polarized images. The method enables production of a high contrast image with well developed grain boundaries, and hence the accuracy of measurements is highly increased. The analysis method introduced has an interest, particularly when microstructures with exaggerated grains are evaluated.

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