INVESTIGATION OF LOCAL TEXTURES IN EXTRUDED MAGNESIUM BY SYNCHROTRON RADIATION

Heinz-Günter Brokmeier (1), Anke Günther (1), Sang-Bong Yi (1), Wenhai Ye (1), Thomas Lippmann (2) & Ulf Garbe (2)

(1) Institute of Materials Engineering, Technical University Clausthal and GKSS-Research Center, Max-Planck-Straße, D-21502 Geesthacht, Germany
(2) HASYLAB at DESY, Notkestraße 85, D-22603 Hamburg, Germany

ABSTRACT

Extruded magnesium was investigated to study the texture variation along the cross section of round and rectangular extruded samples. Our experiments were carried out at the high-field wiggler beam line BW5 (HASYLAB at DESY in Hamburg, Germany). The penetration power of 100 keV synchrotron radiation was sufficient to study extruded rods of 14 mm in diameter in transmission mode. It was demonstrated that the texture is inhomogeneous along the cross section, and moreover that the global texture has a lower degree of preferred orientation than the local texture.

INTRODUCTION

Among others texture is an excellent tool to describe the anisotropy of material properties. Furthermore, the texture indicates also the influence of processing parameters. On one hand the global texture is of special interest because the global texture can be directly related to characteristic material’s parameters obtained by material testing. On the other hand, the local texture shows more details about the complexity of the material. The investigated sample volume can be neglected only in the very rare case of a homogeneous texture along the whole sample. The challenge on the global texture measurement is the averaging over a relatively large sample volume which is comparable to samples for material testing such as tensile samples. Up to now neutron diffraction is the standard method for global texture measurements [1]. Depending on a similar penetration power of high energy synchrotron radiation, this method can also be used for global texture measurements [2-5]. Additionally, a much higher potential of high energy synchrotron radiation is given for local textures. In the case of local texture measurements, a defined relatively small sample volume must be investigated. The current investigation yields with a resolution of about 1 mm³ volume to describe local textures. In the case of synchrotron radiation the volume fraction which can be investigated depends on the grain size and on the availability of very precise slit systems. Moreover, the high brilliance of the synchrotron beam even allows to separate individual grains. Consequently the synchrotron beam can be used as a three-dimensional X-ray microscope [6]. The interest in magnesium and magnesium alloys is based on their potential as light materials for air-space, aviation, and car industry applications. For example, Volkswagen is constructing the prototype of a weight reduced car (about 290 kg) with a gasoline consumption of only one liter per 100 km. Magnesium is similar mostly to aluminum, but due to its hexagonal crystal structure it behaves different. For hexagonal materials the general knowledge is small and only focused on titanium and aluminum. Due to different c/a ratios, these results cannot be transferred to Mg-alloys. Even different types of magnesium alloys
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behave differently caused by the influence of allying elements on the microstructure and the texture. This paper focuses on AZ31 and the texture variation in an extruded rod.

SAMPLE DESCRIPTION

The samples we are interested in are extruded magnesium alloys and magnesium reinforced composites. In this paper we focus on a round extruded piece of a AZ31 alloy. Magnesium powder of 30 µm in grain size was pre-compacted to a cylinder of 74 mm in diameter. Thereafter, this cylinder was hot extruded at about 300 °C with a final strain of 94.1% to a rod of 14 mm in diameter. Due to the high penetration power of thermal neutrons and of high-energy synchrotron radiations the global texture can be measured in a non-destructive way (fig. 1). In order to get the texture variation along the rod cross section the sample was cut to a bar of 3 x 5 x 14 mm³. The whole equipment for texture measurements including the Eulerian cradle was mounted on a z-stage to measure the texture of different positions.

Figure 1: Extruded rod

Figure 2: Samples prepared for global and local texture measurements
EXPERIMENT

The experiments were carried out at the high-field wiggler beam line BW5 (HASYLAB at DESY in Hamburg, Germany). A detailed description of the application of BW5 for texture measurements is given by [3], including also the data evaluation such as corrections and data transfer. In our case we were using an energy of 100 keV corresponding to a wavelength of 0.124 Å. Due to a 1 x 1 mm² slit in front of the sample and the high brilliance of the synchrotron beam we obtain a sufficient local resolution. Looking only on the beam quality the volume fraction could be even much smaller. An area detector (type Mar345 image plate) was used to measure a 2θ range of 5°. A typical exposure obtained in 5 sec. is shown in figure 3.

A number of Debye-Scherrer rings are detected simultaneously. In the case of hexagonal magnesium this number of Bragg-reflections, respectively pole figures, is absolutely sufficient for a quantitative texture analysis. The information of such a 5 sec. shot is:

⇒ There are no single crystal effects that means the grain statistics is good enough (see figure 4).
⇒ The presence of texture is indicated by the varying intensity distribution along individual Debye-Scherrer rings (see figure 5).
⇒ The first three reflections show rather high intensities compared to the outer rings.
⇒ One individual exposure displays a part of the whole set of pole figures

Figure 3: Image plate exposure of a Mg-rod

Figure 4: Sum spectra calculated from figure 3

Figure 5: Intensity distribution along the Mg (100) reflection of figure 3
In order to get the complete texture one needs a set of individual measurements of a rotating sample as described in the past for the film techniques, e.g. in [7-9]. In the presented study we exposed the rotated sample every two degrees from $-90^\circ$ to $+90^\circ$ omega to the synchrotron radiation beam, receiving about 1GB data for a quantitative texture analysis for only one sample position in z-direction. Data evaluation includes corrections of primary intensity, absorption and exposed volume. For detailed information see [10], [3] and [9].

RESULTS

The global texture consists of a double fiber with a strong (100) component, and a much weaker (110) component obtained by neutron diffraction [11]. Figure 7 shows the orientation density of a complete Mg (100) pole figure measured by high energy synchrotron radiation. Firstly, it has to be noticed that in the case of round samples complete pole figures are obtained by both methods – high energy synchrotron radiation as well as thermal neutrons. Secondly, the results of both measurements are surprisingly similar concerning the texture sharpness although pole figure window, counting grid, and data treatment are different. The local textures of volume fractions of about 3 mm$^3$ are stronger than the global texture indicating a texture inhomogeneity. On the sample surface (sample Mg1-0) the strongest texture is observed consisting of the same components than the global texture (fig. 8). In this surface texture the (100) component is much stronger than the (110) component. Moving to the center of the rod (sample Mg1-4) the degree of the (100) component decreases and the (110) component disappears. Contemporaneously another very weak component grows but cannot be explained up to now. Looking on the (002) pole figure the texture variation can be seen as a shift of the Mg (002) peak. In order to detect this peak shift a high resolution, respectively a low pole figure window, is necessary. As shown in figure 10 this peak shift goes from $90^\circ$ (sample Mg1-0) to $85^\circ$ (sample Mg1-4). Due to the high texture symmetry the results given in fig. 10 represent the so called linear pole figure.
CONCLUSIONS

First of all it has to be noticed that the high-energy synchrotron beam has a great potential for a large variety of samples. Local texture measurements to study texture inhomogeneities are possible for much smaller sample volumes compared to neutron diffraction. One of the restrictions is the grain size which didn’t exist in the example presented here. Due to the small pole figure window we are able to get the texture in more detail (see figure 10).

The texture itself shows a gradient with a stronger texture at the rod surface than in the core of the extruded sample. That indicates an inhomogeneous deformation field. A peak shift of the (002) texture peak could result from locally different activations of slip systems or twinning which has to be verified by additional experiments and moreover by texture simulations. Texture
simulations will be able to explain the second texture component which is present in the core of the extruded rod.

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