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Report on the PhD thesis by Krzysztof GŁOWIŃSKI
« Methods for quantitative characterization of three-dimensional grain boundary networks in polycrystalline materials »

M. Krzysztof GŁOWIŃSKI has prepared his PhD at the Institute of Metallurgy and Materials Science (Polish Academy of Science, Krakow, Poland) under the guidance of Adam MORAWIEC. The thesis concludes an extensive work dedicated to the quantitative analysis of three-dimensional grain boundary networks in polycrystalline materials. The experimental techniques allowing for measurement of 3D grain boundary networks have made significant progresses in the past few years and an increasing number of research institutions are currently able to produce such data sets. There is a crucial need for developing and spreading relevant methods for analyzing such datasets. The topic of the PhD itself meets therefore a very actual need of the community, and the quality and relevance of the work done by Krzysztof GŁOWIŃSKI makes his PhD be an important contribution to the field.

The thesis (100 pages only) is written in English and is very well written. The writing is concise, accurate and complete together, which makes the reading of the document very pleasant.

The work comprises complementary aspects:

- Detailed analysis of the tilt/twist character of grain boundaries, using a new parameter for describing the closeness of a grain boundary to a given special configuration. Krzysztof GŁOWIŃSKI confirms the conclusions formerly drawn by his PhD advisor (notably that all symmetry equivalents must be taken into account and not only the disorientation) but he proposes a new parameter much faster to be computed.

Because of the shorter calculation times, the new method appears then as better suited for ‘routine’ analyses.

- Development of a new method for computing grain boundary and grain boundary plane distributions from experimental discrete data, based on a kernel density estimation method. This method allows for avoiding artifacts induced by the previously used binning method, provides much more accurate distributions (in terms of location of the high densities in the five grain-boundary-parameter space) and, in addition, provides an error estimate for densities. Several data sets provided by other research labs (pure nickel, IN100 nickel base superalloy and ferrite) were analyzed. Those data sets had been analyzed previously, so that the performances of the new and old methods could be compared. The improvement is spectacular, the new method allows for depicting contributions that could not be seen with the old method, and for separating peaks which were merged before because of the poor resolution. Furthermore, the error estimate allows for discriminating the peaks that are statistically significant, and therefore should be physically interpreted, from those that are simply arising from statistical fluctuations.
- Implementation of these new tools and methods in a software (GBToolbox) that is freely available, as well as charts indicating the location of geometrically characteristic configurations for CSL boundaries within the cubic crystal symmetry. The development of the experimental techniques for getting three-dimensional information on microstructures and grains boundaries led to the need of suitable analysis tools. No doubt that the community will be grateful to get such relevant and powerful methods implemented in a freeware.
- Application of the proposed methods to datasets from real materials (listed above) and comparison with the performances of the pre-existing methods. Here the reviewer would like to mention a slight shortcoming of the work. The density distributions are analyzed with reference to the random distribution, but the author does not check (or he did but does not mention) if the crystallographic textures of the investigated samples could indeed be considered as close to random. If the crystallite orientations are not random, the misorientation distribution cannot be random. Then the reference should be calculated from the actual texture to check if some of the grain boundary configurations are topologically favored. Furthermore, the high occurrence of some of the boundaries is discussed in relation with the grain boundary energy landscape, but then, the author assumes that the microstructures were at, or close to, equilibrium. For example, some of the grains of the sample Ni (UGent) seem to have internal orientation fluctuations (color gradients on Fig. A.1.b), which does not sound consistent with the result of a prolonged grain growth annealing (necessary for equilibrating grain boundaries). This is not drastically detrimental to the overall quality of the work, but the author could have discussed those physical aspects into more details.

M. Krzysztof GŁOWIŃSKI demonstrates a very good command of mathematics and computer programing, and also the ability of reasonably interpreting his results in terms of grain boundary distributions in polycrystalline materials, which requires also knowledge in materials science.

Furthermore, it must be emphasized that M. Krzysztof GŁOWIŃSKI has published seven journal papers in the last three years (2012 – 2015), which is an exceptional publication rate. In addition, he has published two of these papers alone (the five others being co-signed by his PhD advisor). This is again exceptional and suggests that his own contribution to this work is very significant.

Concluding, the PhD thesis presented by M. Krzysztof GŁOWIŃSKI summarizes an extensive piece of work, and is presented in a very efficient form. The work not only contributes to a better description of the three-dimensional grain boundary networks in the analyzed data sets, but also makes the developed tools available to the community within a freely-distributed software. This work can be ranked as outstanding, and definitely meets the requirements for a defense in view of obtaining the Ph.D. degree.

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