THREE DIMENSIONAL ANALYSIS OF MICROSTRUCTURES

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When using traditional metallographic techniques, materials scientists often make assumptions about the shape, distribution and connectivity of three-dimensional features that lie buried within the material. Recent work has shown that these assumptions can be incorrect. An approach to three-dimensional analysis of microstructures is demonstrated here in three different alloy steels. Proeutectoid cementite, ferrite and an entire pearlite colony were characterized using computer-aided visualization of three-dimensional reconstructions from serial section images. The present paper describes various experimental techniques, as well as recent results and advances in computer-aided reconstruction and visualization.

Introduction

The variety of three-dimensional visualization techniques used in medicine offers a testament to scientists' need and desire to reveal hidden internal structures and connectivities. In traditional metallography, observations of single polished and etched sections allow us to make certain assumptions about the shapes of three-dimensional features that lie buried within a material. Granted, the shapes of many simple features can be accurately deduced in this manner. However, the sizes, spatial distributions, shapes and interconnectivities of complex microstructural features can only be characterized with three-dimensional analysis [1].

The history of three-dimensional analysis in metallography spans at least from 1918 [2] with Forsman's repeated (serial) sectioning effort to understand the three-dimensional structure of pearlite. By projecting the images of each section onto cardboard layers of appropriate thickness, solid models of cementite lamellae were constructed. The past 40 years have seen dramatic improvements in three-dimensional analysis. In 1962, M. Hillert [3] and N. Lange produced a motion picture of serial sections to show the true three-dimensional structure of an entire pearlite colony. Eichen et al. (1964) studied the growth of Widmanstätten ferrite by measuring the changing length of plates with increasing depth through serial sections [4]. Hopkins and Kraft (1965) used a unique "cinephotomicrographic recording of the microstructure of a specimen undergoing controlled electrolytic dissolution."[5] Their results were subsequently represented by building a three-dimensional physical model of Plexiglas to show the eutectic fault structure in a Cu-Al alloy. Hawbolt and Brown (1967) used serial sectioning to study the shapes of grain boundary precipitates in an Ag-Al alloy [6]. Barrett and Yust (1967) showed the interconnectivity of voids in a sintered copper powder [7]. Ziolkowski (1985) used a 'mikrotom' to perform a serial sectioning study of grain boundary precipitates in an □/□ brass alloy [8].

Rhines et al. [9] recommended using a magnification sufficient to show several grains simultaneously and sectioning to a depth of about twice the span of the largest grain in about 250 sections. This rule of thumb must take into account the scale of the features being studied and the limitations of the material removal technique being used. The resolution of the final three-dimensional representations in every serial sectioning project to date has been limited by the thickness of each serial section layer. This compromise is due to both the amount of labor involved (the human aspect) and the problems of storing and properly registering the images once they had been obtained (the computer aspect). Solutions for reducing the amount of labor required and increasing resolution in the thickness direction were to microtome [9] or to avoid sectioning altogether [5]. Also, consider that 250 images captured at 640 pixels by 480 pixels resolution (quite low by current standards) result in a three-dimensional image of 75 megabytes, an immense amount of computer memory in 1980 and not insignificant today. Another problem was that there was no computer software available to obtain three-dimensional measurements or to even accurately display three-dimensional images. In most of the above cases, three-dimensional results were represented by hand-drawn sketches, graphical plots of length versus depth or motion pictures. It was at about this point in time that R.T. DeHoff wrote:

> "In its current embryonic state of development, the use of serial sectioning analysis for all but the most rudimentary of measurements is prohibitively expensive and tedious."[1]

Despite these valid objections, DeHoff predicted the development of automatic aids to reduce the "prodigious" amount of labor involved, and foresaw the rapid development of computer software and hardware that has occurred in the past seventeen years. Since then, image processing and 3-D visualization capabilities have improved to a point where storing and representing three-dimensional images are no longer a severe limitation. In 1991, Hull et al. [10] were among the first to use computer software to contain three-dimensional wire-frame drawings of microstructural features, in this case titanium prior beta grain sizes and shapes. Brystrzycki and Przetakiewicz (1992) used a similar technique to study the sizes and shapes of annealing twins in Ni-2% Mn alloy [11]. A substantial innovative effort was undertaken by H. Wieland, T.N. Rouns and J. Liu (1994) in serial sectioning a recrystallized Al-Mn alloy and simultaneously capturing the crystallographic orientation and location of recrystallized grains using electron backscatter electron diffraction (EBSD) in a scanning electron microscope (SEM) [12]. Three-dimensional representations of data obtained through serial sectioning (either optical, SEM or EBSD) have continued to improve as computer visualization software has advanced [13-18]. Also, recent developments in digital imaging have significantly reduced the efforts required to perform three-dimensional analyses by eliminating the steps to either digitize or manually trace microstructural features. This review is not intended to be complete. There have been many other three-dimensional analysis experiments and many more may have been left unpublished due to the historical difficulty of reproducing three-dimensional representations onto two-dimensional media.

In summary, steady improvements to three-dimensional analysis techniques have resulted in semi-automated material removal, digital image acquisition and visualization of three-dimensional reconstructions using advanced computer software and hardware. The purpose of this paper is to describe an approach to three-dimensional analysis of microstructures that has taken advantage of some of these technological advances. So far, proeutectoid cementite, proeutectoid ferrite and an entire pearlite colony have been characterized using computer-aided visualization of 3-D reconstructions from serial section images. The results of this work demonstrate that three-dimensional analysis can lead to new insights on microstructural evolution.

Experimental Procedures

3-D Analysis of Proeutectoid Cementite. Rapidly solidified specimens of an Fe-13Mn-1.3C alloy were austenitized for 30 seconds at 1100 ˚C in a deoxidized barium chloride salt bath, isothermally reacted at 650 ˚C for 50 seconds and then quenched into iced brine. This produced a structure of proeutectoid grain boundary and Widmanstätten cementite in an austenitic matrix. Rapid solidification reduced the grain size to approximately 25 μm, allowing complete sectioning of entire grains and precipitates while maintaining a fine sectioning increment. A VCR Dimpler was used to perform the polishing procedure to remove approximately 0.2 μm per section. Polished layers were lightly etched with 2% nital. Microhardness indents were used to mark the area of interest, to serve as fiducial marks for subsequent image alignment and to calibrate the depth of material removal. Digital optical micrographs were acquired using a 100X oil immersion objective lens. The images were "registered" with respect to each other in the plane of the image by aligning the hardness indents using Adobe PhotoShop v3.0 and NIH Image v1.61 on a Power Macintosh 7200/120 personal computer. The use of these software products was critical to the success of the present work. PhotoShop was most useful for its editing functions and NIH Image was indispensable for manipulations of image 'stacks'. Stacks of individual digital gray scale images were transformed into 3-D images using AVS version 5.3 visualization software using a Silicon Graphics Onyx workstation. Entire grains as well as individual precipitates were reconstructed by cropping and editing images to contain only the areas of interest. A 'motion picture' sequence of serial sections was also produced. These techniques have been reported previously in greater detail [17].

3-D Analysis of Proeutectoid Ferrite. Homogenized specimens of a high purity Fe-0.12%C-3.28%Ni alloy were austenitized for 30 seconds at 1100 ˚C in a deoxidized barium chloride salt bath, isothermally reacted for 2-3 sec. at 650 ˚C in deoxidized lead baths, and then quenched into iced brine. This produced a structure of proeutectoid grain boundary and Widmanstätten ferrite in a martensitic matrix. The serial sectioning and three-dimensional reconstruction procedures were identical to the above except that a Buehler Minimet was used to polish the material, and picral and nital were used as etchants. A 'motion picture' sequence of serial sections was produced. Individual precipitates were reconstructed by cropping and editing images to contain only regions of interest. Again, these techniques have been reported previously in greater detail [18].

3-D Analysis of Pearlite. Hillert [3] and Lange performed serial sectioning of a pearlite colony formed just below the eutectoid temperature in carburized electrolytic iron. Original optical micrographs were recently supplied by Mats Hillert of the Royal Institute of Technology, in Stockholm, Sweden. The images were scanned at 300 dpi resolution, and registered by matching features such as etch pits and grain boundaries. The variability of contrast and grayscale levels between images required that substantial image processing be performed. The three-dimensional reconstruction procedures were identical to those described earlier. A 'motion picture' sequence of serial sections was produced and individual lamellae were reconstructed by cropping and editing images to contain only information of interest.

Results and Discussion

3-D Analysis of Proeutectoid Cementite. As observed by DeHoff [1], each new alloy system requires different sectioning techniques, especially if the objectives of the study are different. For example, the purpose of the three-dimensional analysis of a Fe-13Mn-1.3C alloy was first to develop experimental procedures for future work, and second to understand the shapes and interconnectivities of proeutectoid cementite precipitates [17]. A typical optical micrograph of this material is shown in Figure 1. In order to accomplish the latter goal, a sectioning increment of 0.2 μm along with a 25 μm average grain size were was selected such that in producing 250 sections a depth of two average grains would be reached. There was significant variability of the sectioning depth (0.17 ± 0.07 μm), due to accumulation of abrasive at the center of the specimen in the VCR Dimpler, even though a 7 mm wide flatting tool was employed.

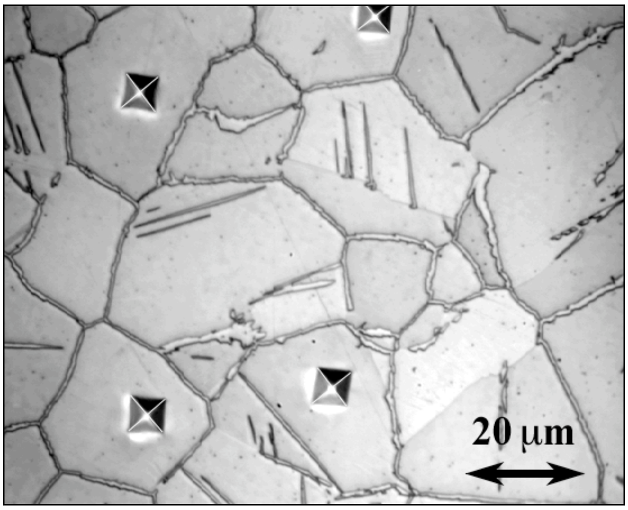

Figure 1 - Typical optical micrograph of an isothermally transformed Fe-13Mn-1.3%C alloy showing proeutectoid cementite precipitates [17].

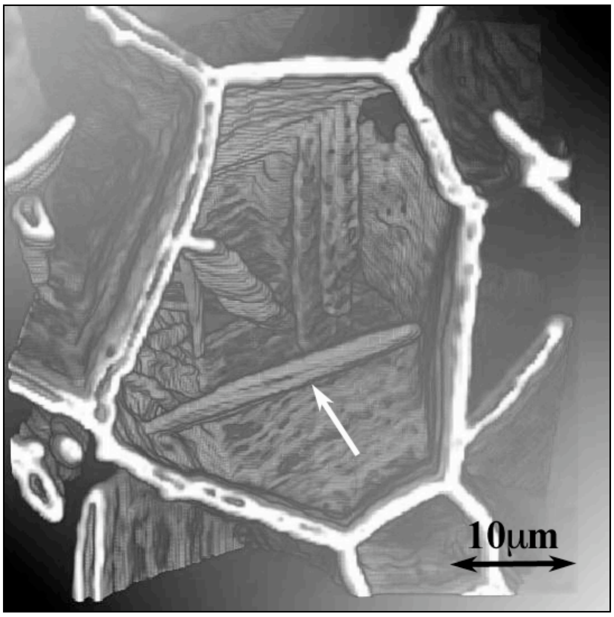

Figure 2 - Three-dimensional reconstruction of proeutectoid cementite precipitates in an isothermally transformed Fe-13Mn-1.3%C alloy [17].

Also, even with light etching, etch pits were often formed over the course of 250 sections, making subsequent image processing more difficult. Nevertheless, at least 20 entire grains and over 200 precipitates were entirely sectioned. A portion of a representative grain is shown in Figure 2 and a precipitate selected from this grain is shown in Figure 3. The ability to digitally remove individual precipitates for study enabled the measurement of each precipitate in three dimensions. Among other results, it was observed that all precipitates were either connected to an austenite grain boundary or another cementite precipitate. Also, in addition to grain boundary precipitates there appeared to be only two distinct types of Widmanstätten precipitates, those with relatively large length-to-width aspect ratios made up of several sub-units (lath-like) and those with relatively small aspect ratios (plate-like). Thus the Dubé morphological classification system [19] was simplified from nine types to only three for proeutectoid cementite.

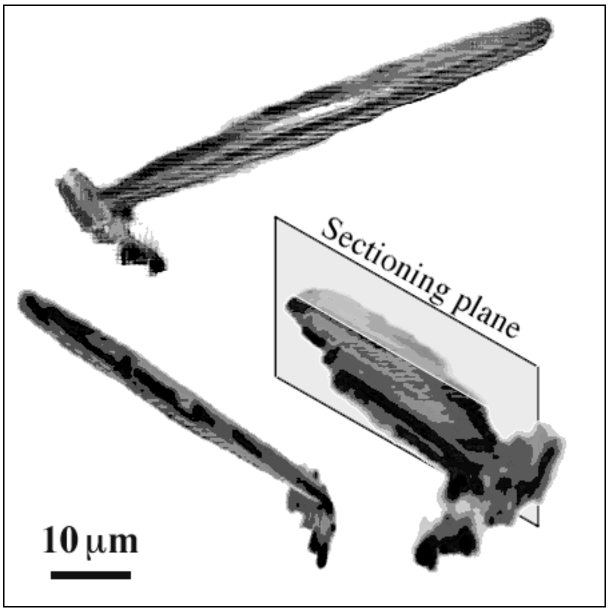

Figure 3 - Three perspective views of the Widmanstätten cementite precipitate indicated by an arrow in Figure 1.

3-D Analysis of Proeutectoid Ferrite. A three-dimensional study of proeutectoid ferrite precipitates [18] revealed a different set of experimental challenges. Most importantly, there was little difference between the grayscale levels of ferrite:martensite boundaries and martensite:martensite boundaries. Also, the alloy responded variably to the etchant (picral). As a result, the final digital images did not allow automatic thresholding and manual editing on the computer was required. A typical optical micrograph of this material is shown in Figure 4. These effects combined to reduce the number of sections to 125, although this number of sections was sufficient to clearly reveal the shape and interconnectivity of several ferrite grains. It was shown that proeutectoid ferrite precipitates (unlike proeutectoid cementite, which quickly wets austenite grain boundaries almost entirely) were not necessarily all interconnected. Although proeutectoid ferrite forms preferentially at prior austenite grain corners and edges, it is also distributed as isolated primary Widmanstätten sideplates along grain boundaries. The three-dimensional rendering of several ferrite precipitates along a former austenite grain boundary shown in Figure 5 illustrates this observation.

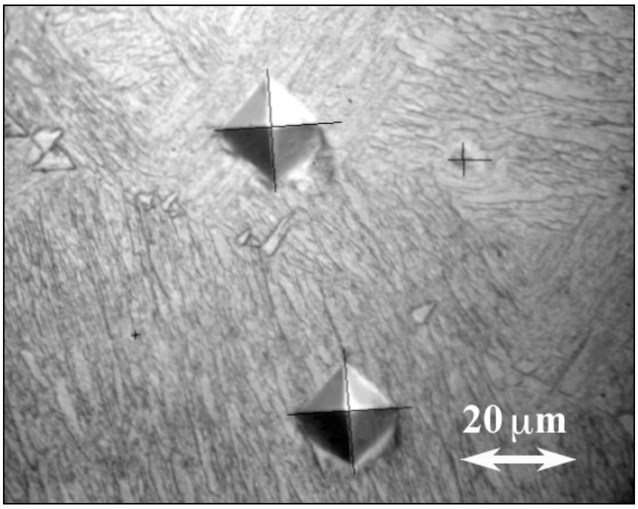

Figure 4 - Typical optical micrograph of an isothermally transformed Fe-3Ni-0.1%C alloy showing proeutectoid ferrite precipitates [18].

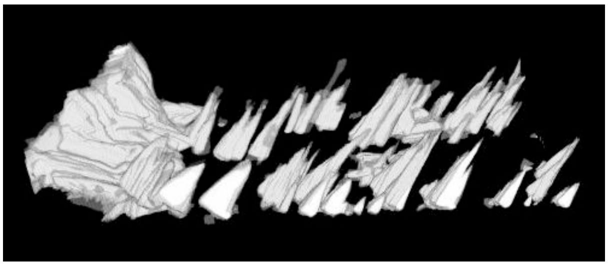

Figure 5 - A three-dimensional reconstruction of the ferrite precipitates shown in Fig. 4, taken from 113 sections spaced 0.3 mm apart in depth [18].

3-D Analysis of Pearlite. Hillert [3] and Lange undertook serial sectioning of an entire pearlite colony to study its true three-dimensional morphology. In the present work, their 241 original micrographs were digitized and processed for three-dimensional reconstruction as shown in Figure 6. Some challenges to the three-dimensional reconstruction of this particular data set were apparent immediately. First, there were no consistent fiducial marks with which to carry out translation/rotation registry between successive images. Occasionally, etch pits could be used to align subsequent images, but when etch pits were not available it was assumed that the colony boundaries and individual lamellae remained in the same relative orientation between slices. Second, there was no way to calibrate exactly the thickness of each section, reported to be approximately 1 micron. Finally, as with all of the materials studied so far, contrast between phases was variable. Extraneous data, such as etch pits, uneven grayscale levels and scratches required manual editing using image processing software. Alternatively, missing boundaries often had to be added. During image processing, some information was lost. For example, although not the focus of this study, the ferrite sub-boundaries arrowed at the top-center of Figure 6a are missing from Figure 6b. Viewing three-dimensional reconstructions of all 241 sections at once has marginal utility because there is too much information to be seen at one time without confusion.

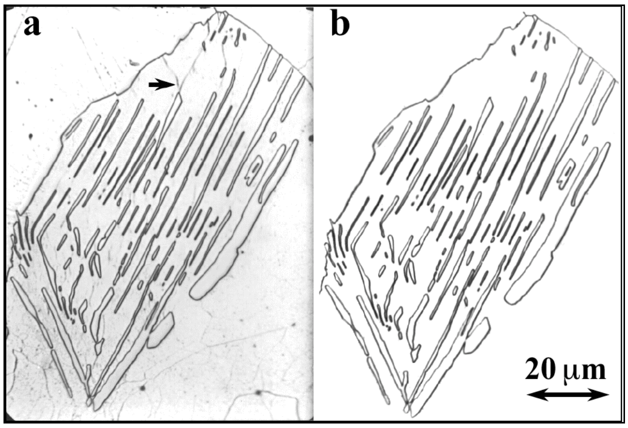

Figure 6 - Original optical micrograph of Hillert and Lange [3] showing an example of the difference between a pre-processed image (a) and a post-processed image (b).

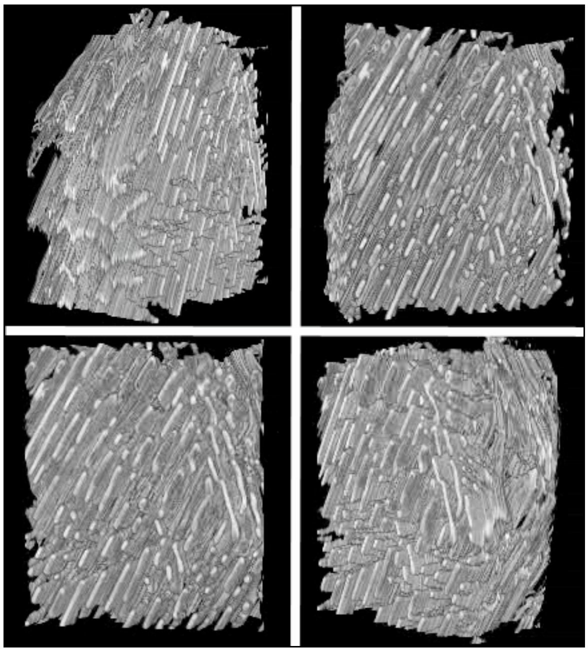

Figure 7 - Viewing several perspectives of a three-dimensional reconstruction of a portion of a pearlite colony aids understanding the three-dimensional morphology.

Reducing the number of slices shown, as well as cropping the dimensions of individual images, result in more meaningful 3-D images such as Figure 7. Even with a reduced data set (cropped from 713 pixels by 851 pixels by 241 sections to 236 by 280 by 160), it becomes difficult to understand the three-dimensional morphology of this colony without viewing multiple perspectives.[*] In the case of pearlite, the three-dimensional shape of a colony is not as much of an issue as the connectivity and shapes of individual lamellae within a colony. Hillert and Lange's work showed that lamellae within a pearlite colony have extensive branching, and are all interconnected. On reviewing any of their 241 optical micrographs individually, there appear to be many isolated cementite precipitates. However, by stepping through the image sequence back and forth, following precipitates as they branch out and intertwine one can see that nearly every portion of the colony is somehow connected to the main branches that bound the lower portions of Figure 6. NIH Image software is so convenient for tracking and marking features directly on the image files that the general interconnectivity of lamellae quickly becomes apparent. However, there are individual lamellae for which the connection is nearly lost due to the relatively coarse sectioning increment such as shown in Figure 8. Smaller precipitates than the one shown in Figure 8 even appear to be disconnected from any other cementite, indicating that the sectioning increment was small relative to the smallest cementite precipitates. In every serial sectioning project, the sectioning thickness must be chosen based on a compromise between the scale of the microstructural features of interest, and the number of sections that can be realistically obtained within a reasonable amount of time. Recent results [20] have confirmed Hillert's conclusions of interconnectivity (which likewise confirmed similar conclusions made by Forsman) by sectioning a smaller, less degenerate pearlite colony with proportionally smaller sectioning increments. Three-dimensional reconstructions of individual lamellae were achieved by simply cropping or editing out all other features. In Figure 9, two parallel lamellae change their apparent habit plane, twisting noticeably about their long dimension axis. Among other possibilities, this apparent twisting could be a result of impingement or interaction with ferrite sub-boundaries. There are many other aspects of this three-dimensional data set, which will continue to be studied.

Related work. The benefits of three-dimensional analysis extend to the results of studies inspired by 3-D observations. For example, subsequent to the three-dimensional analysis of proeutectoid cementite [17], Mangan et al. [21] determined that the two different three-dimensional morphologies of Widmanstätten cementite precipitates correspond to the two known orientation relationships between cementite and austenite. Also, the crystallography of dendritic cementite grain boundary precipitates is the subject of ongoing research to determine whether the primary and secondary dendrite directions are dependent on the orientation of the austenite grain boundary or either austenite grain.

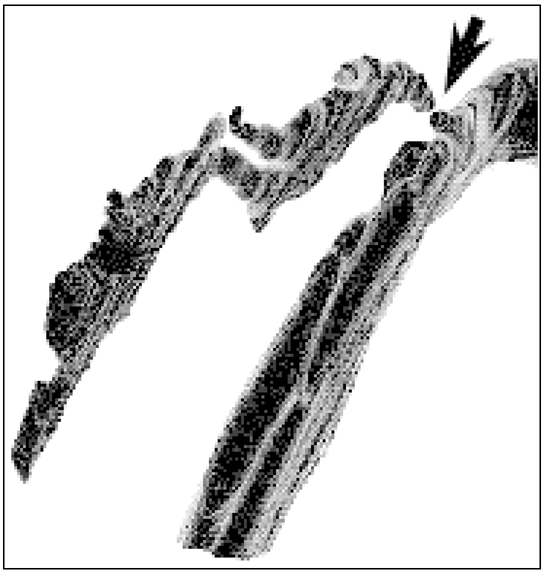

Figure 8 - Three-dimensional reconstruction of cementite lamellae in pearlite. Connections between lamellae are often too small to be resolved easily by optical microscopy.

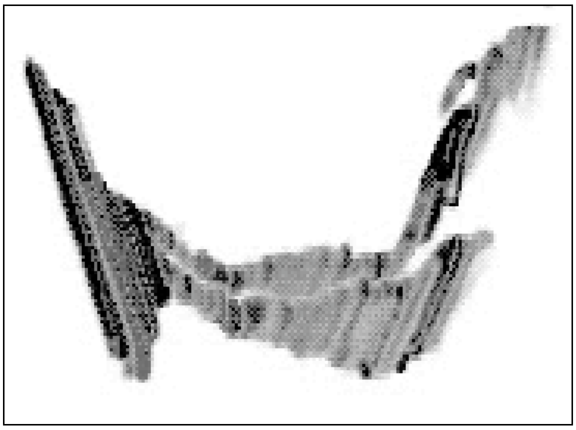

Figure 9 - Three-dimensional reconstruction of cementite lamellae in pearlite. The broad faces of these lamellae twist counter-clockwise from the rear to the front of this perspective view.

Conclusions

The "return on investment" in three-dimensional analysis has improved dramatically with continuous technical and procedural advancements in sectioning, imaging and visualization. The strongest impact has been in the imaging and visualization tools available. Improvement of material removal methods currently offers the most opportunity for making three-dimensional analysis more accessible to materials scientists.

The present three-dimensional analyses have produced important new insights into microstructural evolution, such as

[*] Scale markers are not given for the three-dimensional reconstructions of pearlite because the images have been compressed in the thickness dimension. Each volume picture element (voxel) is 0.29 microns in the x-y plane and 1 micron in the z-direction (the sectioning thickness). The reconstruction software used here requires cubic voxels, therefore the z-dimension was scaled down by a factor of 3.4.

the connectivity and shape of proeutectoid cementite, proeutectoid ferrite and pearlite lamellae. The insights that are gained from three-dimensional analysis not only have immediate rewards, but lead to new avenues of research.

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References

1. R.T. DeHoff, Quantitative serial sectioning analysis: preview. J. Microscopy, 131(3), 259-263 (1983).
2. O. Forsman, Undersökning av rymdstrukturen hos ett kolstål av hypereutektoid sammansättning. Jern-Kontorets Ann., 102, 1-30 (1918).
3. M. Hillert, The Decomposition of Austenite by Diffusional Processes, V. F. Zackay and H. I. Aaronson (editors), Interscience, New York, 197-237 (1962).
4. E. Eichen, H. I. Aaronson, G.M. Pound and R. Trivedi, Thermionic emission microscope study of the formation of ferrite sideplates. Acta Metall., 12, 1298-1301 (1964).
5. R.H. Hopkins and R.W. Kraft, A rapid technique for observation of three-dimensional microstructures. Trans. AIME, 233, 1526-1532 (1965).
6. E. B. Hawbolt and L. C. Brown, Grain boundary precipitation in Ag-5.64 wt pct Al alloys. Trans. AIME, 239, 1916-1924 (1967).
7. L.K. Barrett and C.S. Yust, Progressive shape changes of the void during sintering. Trans. AIME, 239, 1172-1180 (1967).
8. F.N. Rhines, K.R. Craig and D.A. Rousse, Measurement of average grain volume and certain topological parameters by serial section analysis. Metall. Trans, 7A, 1729-1734 (1976).
9. P.M. Ziolkowski, The three dimensional shapes of grain boundary precipitates in □/□ brass. MS Thesis, Mich. Tech. (1985).
10. D.A. Hull, D. McCammond, D.W. Hoeppner and W.G. Hellier, Titanium prior-beta grain volume distribution by quantitative serial sectioning techniques. Materials. Char., 26, 63-71 (1991).
11. J. Brystrzycki and W. Przetakiewicz, 3-Dimensional reconstruction of annealing twins shape in FCC metals by serial sectioning. Scripta Metall. Mater., 27, 893-896 (1992).
12. H. Wieland, T.N. Rouns and J. Liu, Z. Metallkd., The role of particle stimulated nucleation during recrystallization of an aluminum-manganese alloy. 85, 592-597 (1994).
13. M. A. Mangan and G.J. Shiflet, Three-dimensional reconstruction of pearlite colonies. Solid-solid Phase Transformations Proceedings, held at Farmington, PA, W.C. Johnson, J.M. Howe, D.E. Laughlin and W.A. Soffa (editors), TMS, Warrendale, PA. 547-552 (1994).
14. S. Matsuoka, M. A. Mangan and G.J. Shiflet, Morphological development of cellular colonies in a 19Cr-5Ni austenite steel. Solid-solid Phase Transformations Proceedings held at Farmington, PA, W.C. Johnson, J.M. Howe, D.E. Laughlin and W.A. Soffa (editors), TMS, Warrendale, PA 521-526 (1994).
15. M. A. Mangan, P.D. Lauren and G.J. Shiflet, Three-dimensional reconstruction of Widmanstätten plates in Fe-12.3Mn-0.8C. J. Micros., 188(1), 36-41 (1997).
16. T.L. Wolfsdorf, W.H. Bender and P.W. Voorhees, The morphology of high volume fraction solid-liquid mixtures: an application of microstructural tomography. Acta Mater., 45(6), 2279-2295 (1997).
17. M.V. Kral and G. Spanos, Three-dimensional analysis of proeutectoid cementite precipitates. Acta Mater., 47(2), 711-724 (1999).
18. G. Spanos and M.V. Kral, . Three dimensional reconstruction and classification of ferrite precipitates. In: Koiwa M, Otsuka K, Miyazaki T. editors. International Conference On Solid-Solid Phase Transformations '99. Kyoto, Japan. Japan Institute of Metals, 1999: 1497-1500.
19. C. A. Dubé, H. I. Aaronson and R. F. Mehl, La formation de la ferrite proeutectoïde dans les aciers au carbone. Rev. Met., 55, 201 (1958).
20. M. A. Mangan and G.J. Shiflet, Pitsch-Petch orientation relationship in ferrous pearlite at small undercooling. Metallurgical and Materials Transactions A v30 n11, 1999: 2767-2781.
21. Mangan, M.A., Kral, M.V. & Spanos, G. Crystallography and Morphology of Cementite Precipitates. *Acta Materialia*, Vol. 47no17, 1999: 4263-4274.