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Three-dimensional analysis of coalesced bainite using FIB tomography

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Abstract

A coarse grained constituent recently discovered in high strength steel weld metals – Coalesced Bainite – was investigated with a serial section procedure for three-dimensional (3D) analysis using Focused Ion Beam (FIB) milling. Image planes of a coalesced bainite grain, 0.4 μm apart were acquired using the FIB. The data collected was then used to create a 3D model of the coalesced bainite grain. The reconstruction, revealed the grain was large chunky in all three dimensions which was greater than 20 μm in length. Correlations were made between the 3D reconstruction and micrographs of the same coalesced bainite grain recorded using Field Emission Gun Scanning Electron Microscopy and Light Optical Microscopy to allow an understanding in 3D when making future interpretations using the latter characterisation techniques.

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Introduction

It has been observed that lower bainitic and lath martensitic microstructures sometimes form with a bimodal size distribution of plates and are often accompanied with large platelets that can be observed optically [1-5]. A detailed study of these large plates was undertaken in the mid 1990's and it was reported that they form as a result of a coalescence of bainitic platelets when the bainite formation temperature is close to the martensite start temperature [5]. More recently these large grains of — coalesced bainite — were characterised and found to develop in experimental high strength steel weld metals [6-11]. Some typical micrographs obtained using field emission gun scanning electron microscopy (FEGSEM) that show coalesced bainite in high strength steel weld metals are presented in Figure 1.

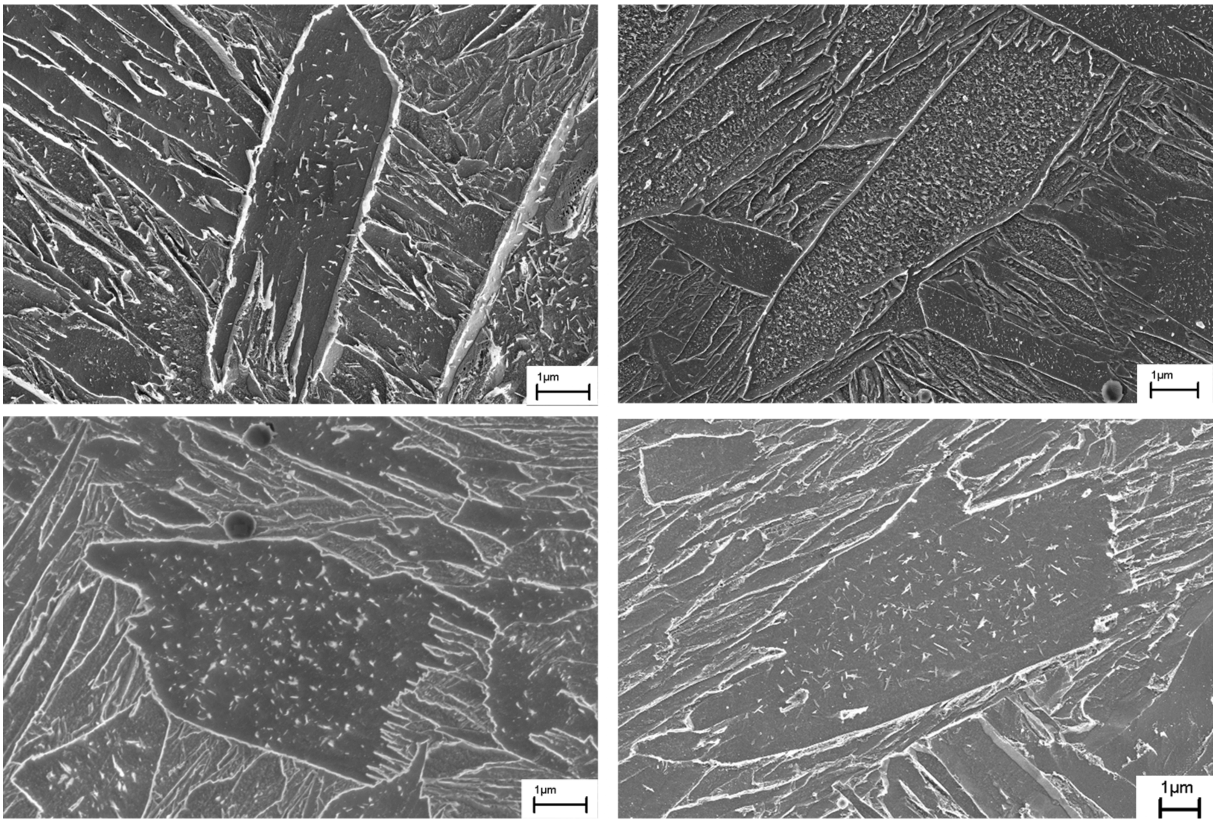

Figure 1 The typical morphology of coalesced bainite in different high strength steel weld metals shown using FEGSEM.

In all work to date, light optical microscopy (LOM), FEGSEM or TEM was used to image and characterise coalesced bainite. These characterisation methods can only reveal two-dimensional (2D) or topographic information from sections or surfaces. Consequently, the metallurgist is left to ponder, make interpretations based on 2D imaging and subsequently a desire for three-dimensional (3D) characterisation arises.

Focused ion beam (FIB) tomography allows the 3D reconstruction of microstructural features with resolution less than 0.1 μm attainable [12, 13]. FIB tomography is based on a serial sectioning procedure where a series of layers with constant thickness in the z-direction are milled away using the ion beam. Between the removal of each layer, an image is recorded and stored. These images are then transferred into a voxel (volume pixel) based data volume and post-processed using computer software. Since the size of the coalesced bainite plates were deemed to be of particular importance for the mechanical properties of high strength steel weld metals it was decided to apply this relatively new microstructural characterisation method in order to get a three dimensional view.

Sample Preparation

A weld alloy whose composition (Table 1) was specifically designed with the aim of producing large amounts of coalesced bainite was chosen for investigation. The welded joint was produced using shielded metal arc welding and previous metallographic studies confirmed that significant amounts of coalesced bainite formed within the weld metal **[14]**.

Table 1 Weld metal composition and welding parameters. The composition in wt. % unless stated along with welding parameters interpass temperature (°C) and heat input (kJ/mm).

C	Ni	Mn	Cr	Mo	Si	P
0.079	10.6	0.57	1.12	0.3	0.24	0.011
Cu	S	O (ppm)	N (ppm)	V	Interpass temp	Heat input ($kJmm^{-1}$)
0.30	0.008	360	110	0.006	200°C	1.2

A specimen from the weld metal cross section was extracted perpendicular to the welding direction, mounted in bakelite, wet ground, polished to 1 µm diamond solution and etched using 2 % nital etchant. To allow the transfer of knowledge between different characterization methods an area of interest was first identified and investigated with LOM (Figure 2a). A hardness indent was place close to the region to allow easy tracking when moving between different instruments. The same area was also investigated with FEGSEM in the secondary electron mode (Figure 2b). A Leitz Aristomet light optical microscope and a Leo Ultra 55 FEGSEM were used in these examinations.

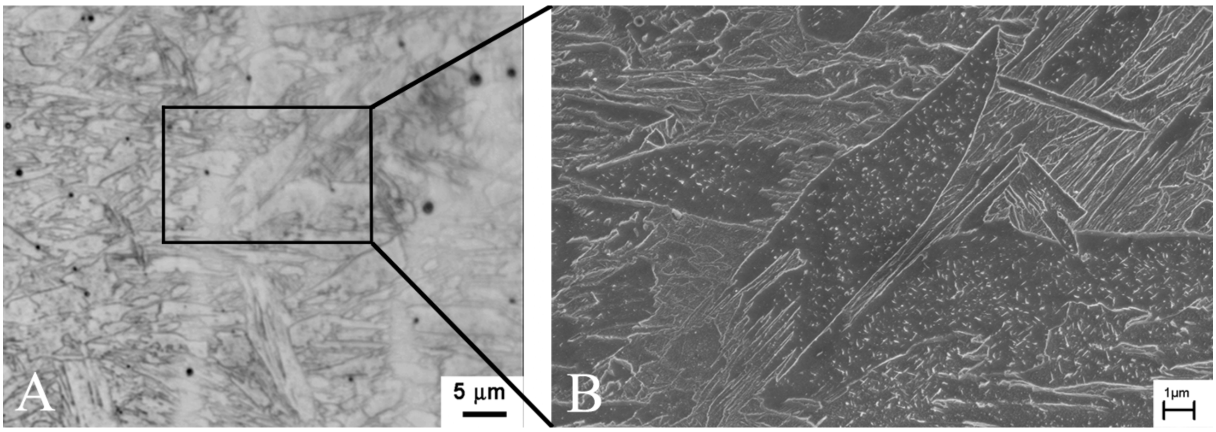

Figure 2 LOM (A) and FEGSEM (B) micrographs showing the grain of coalesced bainite selected for FIB tomography studies.

Cube Preparation for FIB tomography

In order to carry out the serial sectioning procedure using the FIB, a cube incorporating the area of interest, first needed to be prepared. Figure 3A shows an ion image of the same coalesced bainite grain as in Figure 2 that was investigated. In order to protect the grain from undesired ion induced erosion when imaging a Pt layer was first deposited over the area of interest (Figure 3B). To allow imaging in the X-Y plane, a trench had to be milled away in front of the cube (Figure 3B). Additional trenches were made on each side of the cube in order to eliminate shadowing effects and to avoid the excess redeposition of material in front of the X-Y plane that would result in difficulties when imaging (Figure 3C). Finally, a fine milling / polishing was carried out with a low ion beam current around the cube edges to enhance image contrast (Figure 3D).

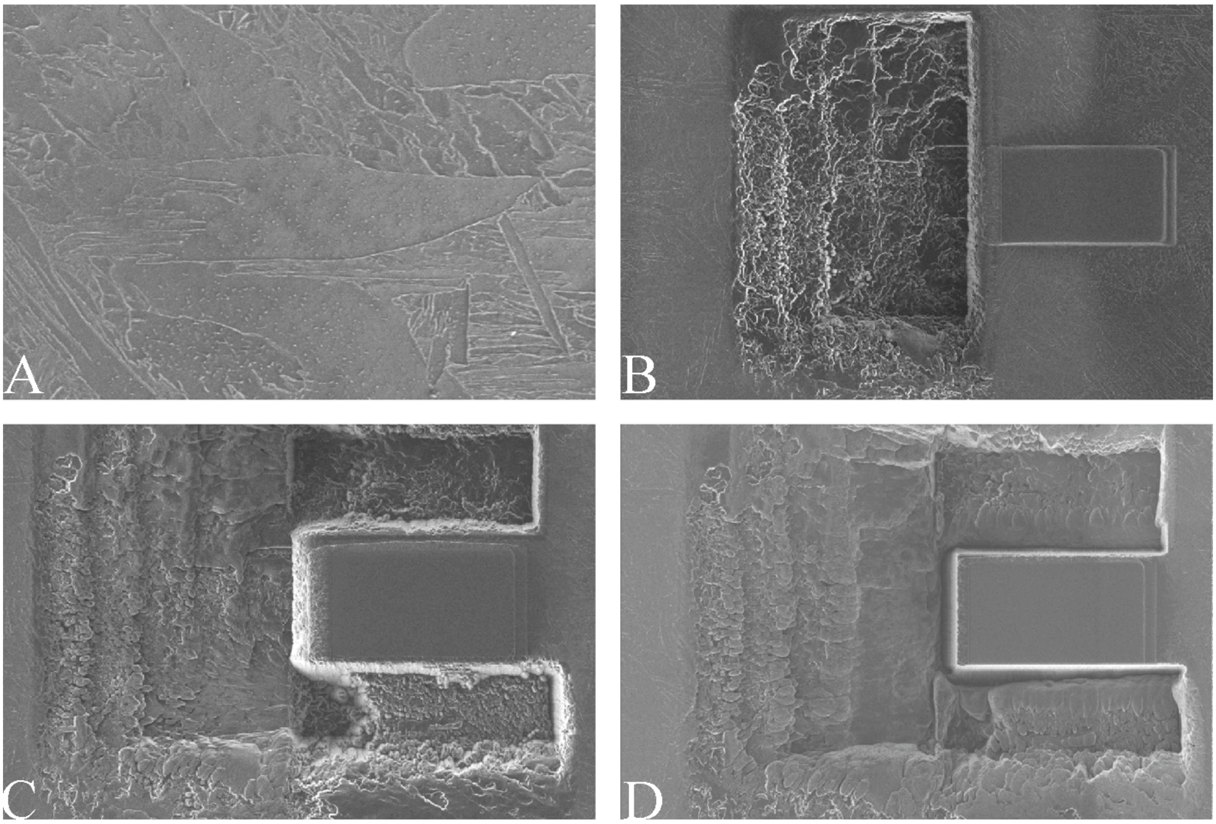

Figure 3 FIB micrographs showing different stages in area preparation. **3A,** an ion beam image of the coalesced bainite grain. **3B,** Pt has been deposited over the area of interest and initial milling of the trenches has been started. **3C,** all trenches surrounding the area of interest has been milled out. **3D,** fine polishing of the cube edges has been carried out.

Once the cube was prepared, the serial sectioning procedure could be carried out. The specimen was placed at eucentric height to allow for the cube to be in the same position after tilting, before and after each sectioning and imaging step was carried out. Since the coalesced bainite grain was so large (14 μm long and 4 μm wide, Figure 2) a balance between different settings had to be found when carrying out the work. The first decision to be made was on the choice of slice thickness. Here 0.4 μm was chosen since it was believed that the coalescence of the individual plates would be captured reasonably well with an estimated 10 slices to be made through that region. The accuracy of the reconstruction could be greatly be improved by choosing a thinner slice thickness when milling but there was a trade off due to the milling time for each slice (25mins) and the overall length of the grain. There was a similar trade off with the choice of beam current used when carrying out the milling. It was found that the lower the beam current used the smoother the surface finish of the XY plane of the sample became leading to higher quality images for the reconstruction purpose. After experimenting a little, an ion beam current of 300 ρA in combination with 30 kV was chosen as the optimum for the milling. For imaging, an ion beam current of 27 ρA with 30 kV was used. For FIB images, material contrast is pronounced due to sensitivity of the secondary ion yield to matrix effects and surface conditions. As a result, it is possible to differentiate between different grains / plates without etching the sample surface. In total 49 slices were removed from the cube with the last 40 containing information about the coalesced bainite grain. Each slice took approximately 25 minutes of milling time.

Results

Data processing and 3D Reconstruction

Figure 4 A, B and C presents three of the 49 ion images at different locations throughout the cube. The three FIB images are correlated "approximately" to the FEGSEM image of the coalesced bainite grain (Figure 4D). In Figure 4D, the FEGSEM micrograph of Figure 2 was rotated so that the Z direction of the coalesced bainite grain is horizontal and can be easily correlated to the FIB images that present the XY plane. The coalesced bainite grain of interest had a dark grey contrast. In Figure 4A the coalesced bainite grain was not yet sectioned though while in Figure 4B the coalesced bainite grain has been entered and is seen just under the Pt layer on the top of the ion image. Measuring the width of the coalesced bainite grain under the Pt layer in the ion images and correlating it to the width across the coalesced bainite grain in the FEGSEM image allowed the approximate position of the slices to be located.

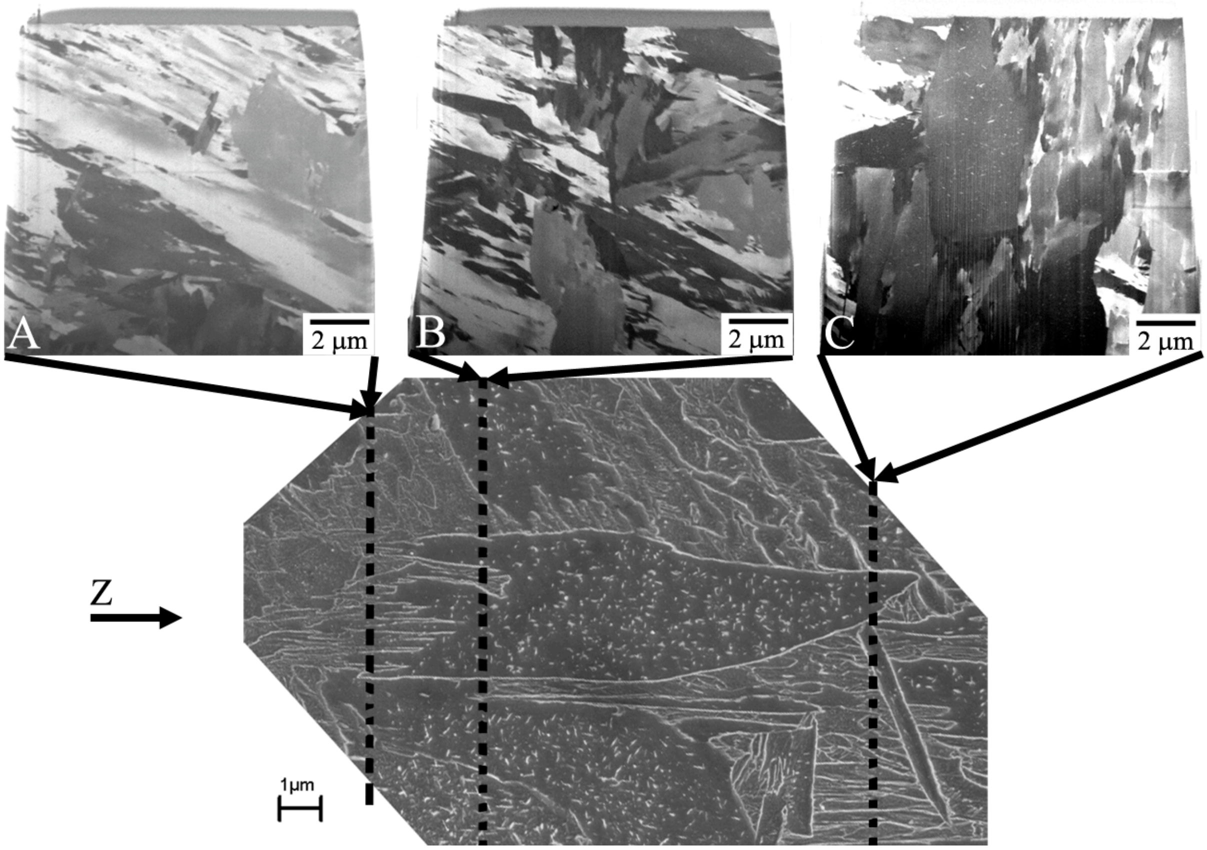

Figure 4 Three FIB images (A-C, xy-direction), taken after various amounts of milling through the cube. The approximate location of the FIB images is related to the FEGSEM image (bottom) that shows the coalesced bainite grain in the xz-direction.

To allow the reconstruction of the coalesced bainite in three dimensions the stack of 49 images were entered into the Amira 4.1 software package [15] and transformed into a voxel-based data volume. This volume was used as the basis for all subsequent computational analysis. The images were first overlay aligned using a reference marker and entered into a bounding box. The coalesced bainite grain was then highlighted on each image using the detail image segmentation options. In 3D reconstruction, segmentation of a given detail is where the most uncertainty and potential error lies. It is of critical importance to have sharp primary images with high resolution, good contrast and low noise for accurate results. On completion of segmentation, the images were further processed and a triangulated surface was constructed within the bounding box. Figure 5 presents six snap shots of the coalesced bainite reconstruction that can be related to the 2 dimensional FEGSEM image of the coalesced bainite grain investigated. Using the software, it was possible to rotate the bounding box containing the reconstruction in any of, or a combination of, the X, Y and Z directions and to visualise the grain in 3D. It should be noted that the snap shots of the coalesced bainite reconstruction were not assigned a scale.

It is observed that the coalesced bainite grain is a large, chunky grain. From a combination of the FEGSEM and FIB micrographs (Figure 4 and 5) it is seen that the portion of the coalesced bainite grain extending below the surface of the specimen had a width up to the region of 4 μm (x-direction), a depth greater that 6 μm (y-direction) and length greater than 20 (z-direction). In addition, it was possible to visualise the grain in three dimensions within the microstructure from the reconstruction.

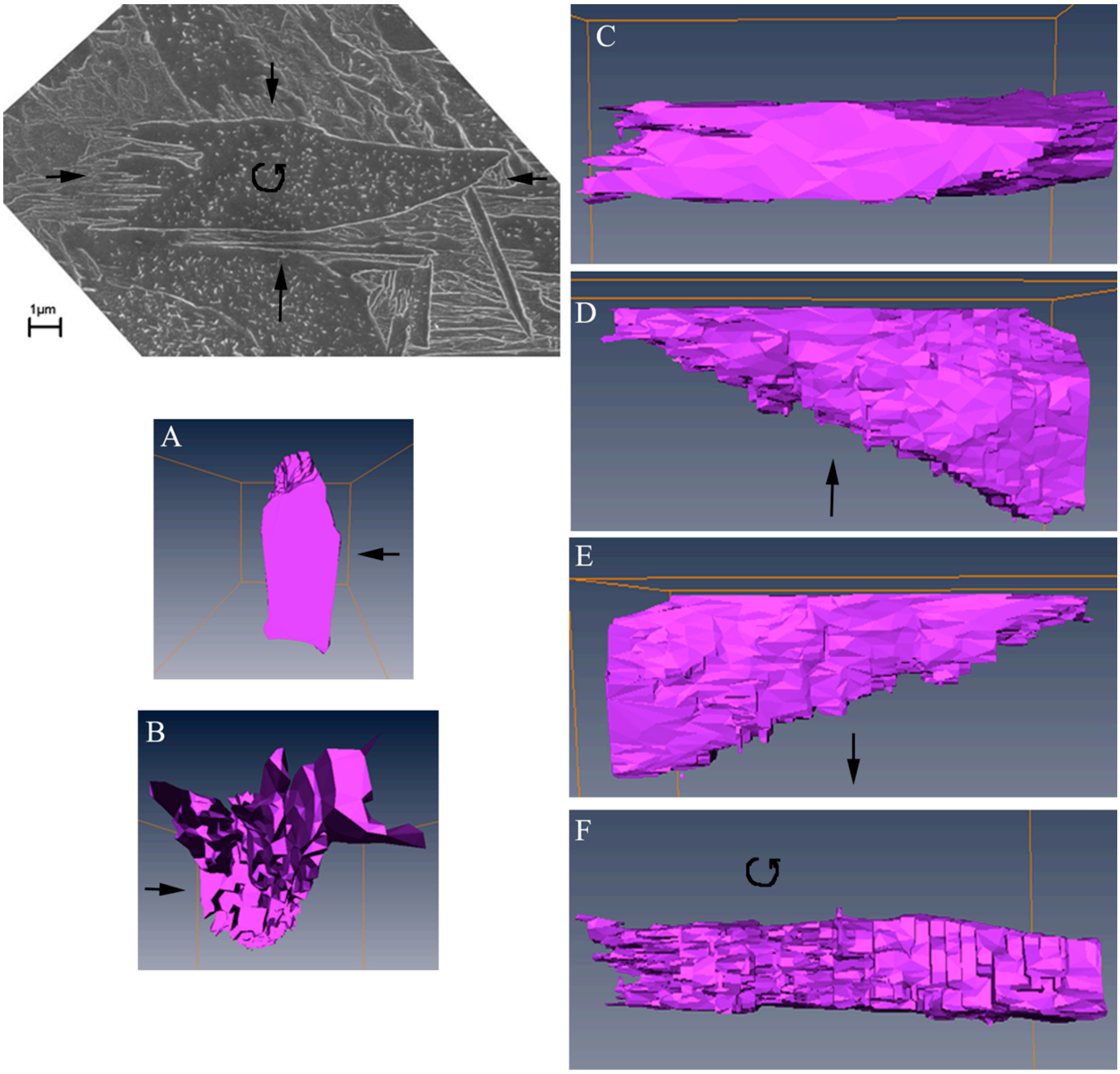

Figure 5 Six snap shots of the 3D coalesced bainite reconstruction that was made using Amira 4.1 software [15] along with the corresponding FEGSEM of the same grain. The arrow in each snap-shot of the 3D reconstruction is related to the same arrow type in the FEGSEM image and indicates what direction the coalesced bainite grain was viewed from. "→" is front view, "↓" left side view "←" rear view, "↑" right side view, "□" view from underneath. Image C is the top view as reconstructed using the Amira software.

Discussion

The experimental work presented in this paper was carried out in a systematic fashion with the same area investigated with LOM, FEGSEM and FIB. As a result, correlations can be made between the 3D reconstruction (obtained using the FIB) and micrographs from the FEGSEM or LOM. This transfer of knowledge between the different characterisation microscopes is powerful in allowing a metallurgist visualise the microstructure in all three dimensions when working with basic LOM. The fact that the same area / grain was investigated with all instruments also eliminates all questions that normally arise about differences between different grains or areas after examinations have been carried out.

There is excellent agreement between the FEGSEM image and Figure 5C that both show the top view of the coalesced bainite grain. Therefore one can assume that the 3-dimensional reconstruction is in good agreement with the actual shape of the coalesced bainite grain. The coalescence of the individual plates to form the large bainite grain was considering the rather large step size, also reasonably well captured. Looking at the view from underneath (Figure 5F “□”) the reconstruction appears a little jagged. There are a number of reasons for this; first the grain is inclined to the surface and secondly the step size of 0.4μm is too large to capture the inclination smoothly. The jagged underneath surface can also be observed from the side views (Figure 5D and E, “↑” right side view and “↓” left side view).

It is seen that a section, and not the complete coalesced bainite grain, was reconstructed in Figure 5. There are a number reasons for this; first, a cross section of the welded joint was initially taken for the investigation. At this point, a section of the coalesced bainite grain was removed during specimen preparation. Secondly, the sectioning procedure was finished without coming to the end of the grain because the initial area prepared for the experiment (the two side trenches) had come to an end. It was found that the overall length of the grain (z direction) was much larger than first estimated. Despite these shortcomings, it can be concluded that the coalesced bainite grain is large in all three dimensions with a width up to the region of 4 μm (x-direction), a depth greater that 6 μm (y-direction) and a length greater than 20 (z-direction).

The most important finding of this work is that a visual image of how the coalesced bainite grain appears in three dimensions was obtained. It was found that the coalesced bainite grain is much larger than first imaged with FEGSEM and actually extends in the form of a large chunky grain above and below the surface.

Conclusions

The 3D shape of coalesced bainite, a constituent recently discovered in high strength steel weld metals, was examined. A serial section procedure was applied using FIB. Image planes, 0.4 μm apart, were collected and transformed into a 3D data volume. The data then allowed the successful reconstruction of a coalesced bainite grain in three dimensions.

The reconstruction revealed a large coalesced grain in all three dimensions. The coalesced bainite grain extended far below the surface examined with FEGSEM and LOM. It was established that the section of the grain below the specimen surface had a depth greater that 6 μm, a width up to the region of 4 μm and a length greater than 20 μm. Correlations were made between the 3D reconstruction and micrographs taken using FEGSEM and LOM to allow future metallurgist an understanding in 3D when making interpretations using the latter characterisation methods.

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References

1. Bhadeshia, H.K.D.H. and Edmonds, D.V.,1979. *The Bainite Transformation in a Silicon Steel.* Metallurgical Transactions A, **Vol. 10A**: p. 895-907.
2. Bhadeshia, H.K.D.H., *Theory and Significance of Retained Austenite in Steels*. 1979, University of Cambridge.
3. Bhadeshia, H.K.D.H. and Edmonds, D.V.1979. in *Phase Transformations 2, IV-4*. London: Institution of Metallurgist.
4. Padmanabhan, R. and Wood, W.E.,1984. Materials Science and Engineering, **66**: p. 1.
5. Chang, L.C. and Bhadeshia, H.K.D.H.,1996. *Microstructure of lower bainite formed at large undercoolings below bainite start temperature.* Materials Science and Technology, **12**(3): p. 233-236.
6. Keehan, E., Karlsson, L., Andrén, H.-O., and Bhadeshia, H.K.D.H.,2006. *Influence of C, Mn and Ni on Strong Steel Weld Metals: Part 3, Increased Strength from Carbon Additions.* Science and Technology of Welding and Joining, **11** p. 18-24.
7. Keehan, E., Karlsson, L., Andrén, H.-O., and Bhadeshia, H.K.D.H.,2006. *Influence of C, Mn and Ni on Strong Steel Weld Metals: Part 2, Impact Toughness Gain from Manganese Reductions.* Science and Technology of Welding and Joining **11** p. 9-18.
8. Keehan, E., Karlsson, L., and Andrén, H.-O.,2006. *Influence of C, Mn and Ni on Strong Steel Weld Metals: Part 1, Effect of nickel content.* Science and Technology of Welding and Joining, **11**: p. 1-8.
9. Keehan, E. and Karlsson, L.2005. *New alloying concepts for high strength steel weld metals*. in *Super High Strength Steels*. Rome, Italy.
10. Keehan, E., Andrén, H.O., Karlsson, L., and Svensson, L.-E.,2006. *New developments with C-Mn-Ni high strength steel weld metals, Part B. Mechanical Properties.* Accepted to The Welding Journal.
11. Keehan, E., Andrén, H.O., Karlsson, L., and Svensson, L.-E.,2006. *New developments with C–Mn–Ni high strength steel weld metals, Part A. Microstructure.* Accepted to The Welding Journal.
12. HOLZER, L., INDUTNYI, F., GASSER, P., MÜNCH, B., and WEGMANN, M.,2004. *Three-dimensional analysis of porous BaTiO3 ceramics using FIB nanotomography.* Journal of Microscopy, **216**(Pt 1): p. 84-95.
13. Inkson, B.J., Mulvihill, M., and Mobus, G.,2001. *3D determination of grain shape in a FeAl-based nanocomposite by 3D FIB tomography.* Scripta Mater., **47** (7): p. 753-758.
14. Keehan, E., Karlsson, L., Andrén, H.-O., and Bhadeshia, H.K.D.H.2005. *Understanding Mechanical Properties of Novel High Strength Steel Weld Metals Through High-Resolution Microstructural Investigations*. in *International Conference: Trends in Welding Research*. Alanta, Georgia, USA: ASM International.
15. Mercury Computer Systems, I., *Amira*. 2006.