UPDATE ON FRACTURE TOUGHNESS VARIABILITY IN F82H—D. S. Gelles (Pacific Northwest National Laboratory)* and Mikhail A. Sokolov (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this effort is to better understand the fracture toughness response of low activation ferritic steel F82H.

SUMMARY

The fracture toughness database for F82H displays some anomalous behavior associated with the center of the 25 mm thick plate. Metallographic carbide etchant reveals larger particles dispersed through the 25 mm thick F82H plate. The particles are found to be rich in Ta and O. Size distribution measurements indicate no enhancement at the center of the plate. However, the spatial distribution is affected so that large particles are more often located next to other large particles in the center of the plate. A mechanism is proposed that promotes easy crack nucleation between large tantalum oxide particles.

PROGRESS AND STATUS

Introduction

As described previously [1], fracture toughness measurements of 25 mm F82H plate are showing indications of non-uniform behavior as a function of thickness [2]. The purpose of the present effort is to continue to identify the microstructure features that may be responsible for this fracture toughness degradation.

Experimental Procedure

Specimens previously prepared for metallographic examination have been re-etched using Vilella's etch, and examined metallographically and by scanning electron microscopy (using a JEM-840 Scanning Electron Microscope (SEM) operating at 20 KeV equipped with a backscatter detector.) Images were stored and quantified digitally.

Results

Metallography

Both transverse and longitudinal etched samples showed large individual equiaxed particles fairly uniformly distributed. A portion of the transverse section was selected for detailed study and is shown in Figure 1a. The section was approximately 6.8 x 25 mm (the full thickness of the plate). Any large carbides found were photographed with positions recorded. Examples of some of the largest particles found are shown in Figures 1b) through g), with the corresponding micrograph numbers superimposed on Figure 1a) to show the location of each particle. The particles are often slightly elongated in the rolling direction of the plate, are often (but not always) associated with prior-austenite grain boundaries, and are usually isolated away from other particles.

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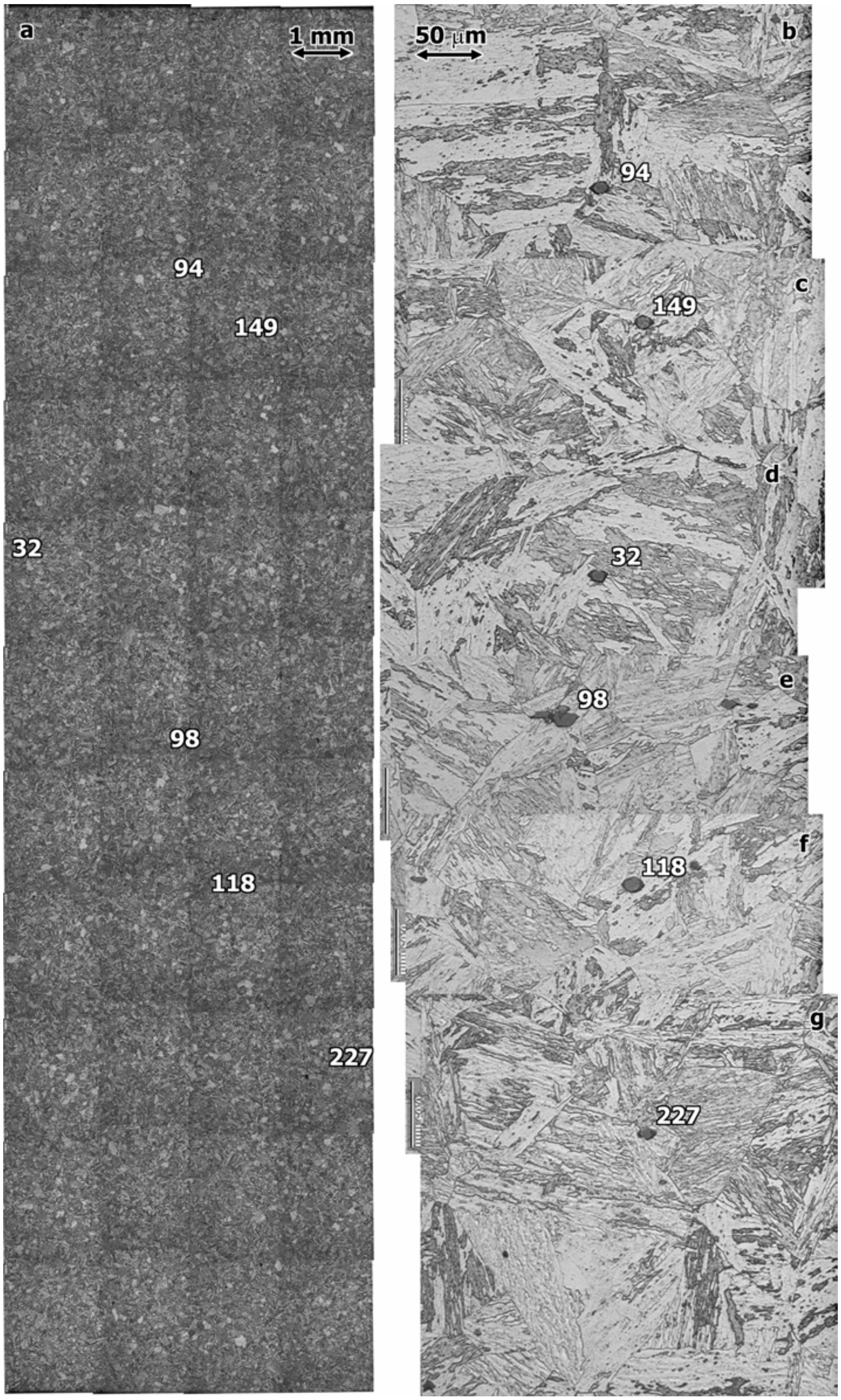

Figure 1. Transverse section etched microstructure of F82H plate showing in a) a mosaic of the microstructure across the full width of the 25 mm plate and in b) through g) some of the largest particles found. The numbers shown refer to the micrograph numbers, with positions indicated in a).

The micrographs containing particles were measured to provide approximate particle diameters for 480 particles, and these particle sizes have been plotted as a function of position from one of the surfaces in order to identify any non-uniformity in the distribution. The plot is shown in Figure 2. From Figure 2 it is apparent the particles range in size form 2 to 12 m with a fairly uniform distribution as a function of distance from the surface. Smaller particles were present but were not measured. It can be argued that few of the larger particles were less than 5 mm from the surface, but at issue is whether an explanation can be found for lower fracture toughness in the middle 5 mm of the plate. Therefore, it is concluded that reduced fracture toughness in the center of the 25 mm plate cannot be ascribed to variations in the size of these large particles.

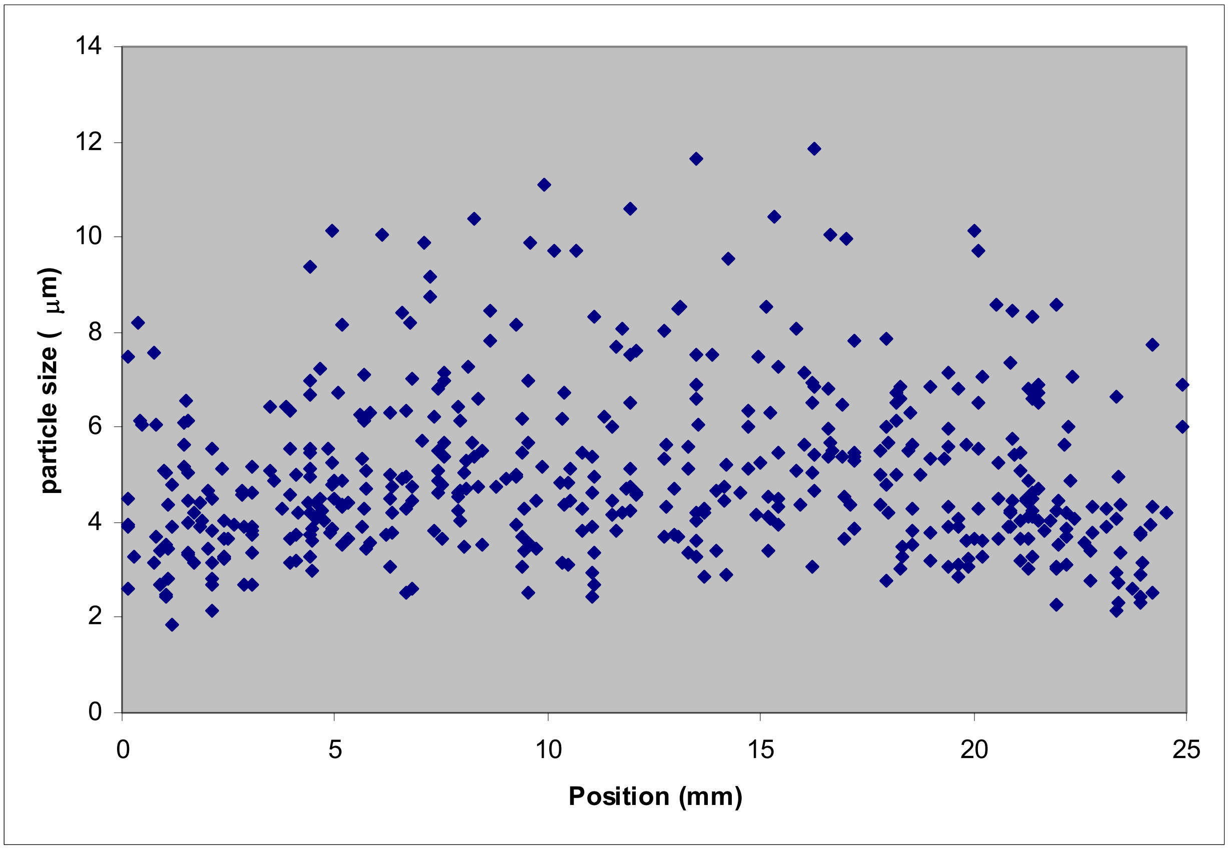

Figure 2. Particle size as a function of position in the 25 mm plate of F82H.

The particle size distribution for particles measured is shown in Figure 3. The largest fraction of the particles was in the range 3.5 to 4.5 µm. Based on a spherical approximation, the areal fraction of particles that were measured in the region shown in Figure 1a) was 0.10%, indicating that the volume fraction for these particles is only 0.1%.

However, in the course of examination, it became apparent that particles tended to clump, but clumping was generally restricted to the center of the plate. This tendency can be noted in Figure 1e) showing a line or stringer of particles 13.5 mm from the top surface. To the left and below the numerals “98”, a clumping of particles can be identified. Other examples of such clumping selected from the entire transverse section sample are provided at higher magnification in Figure 4. Examples 4a), 4e) and 4f) were from the area selected for examination, and therefore the larger particles shown are from outside the area shown in Figure 1a). All were found in the center of the plate. It therefore is apparent that particles had a greater tendency for clumping in the center of the plate.

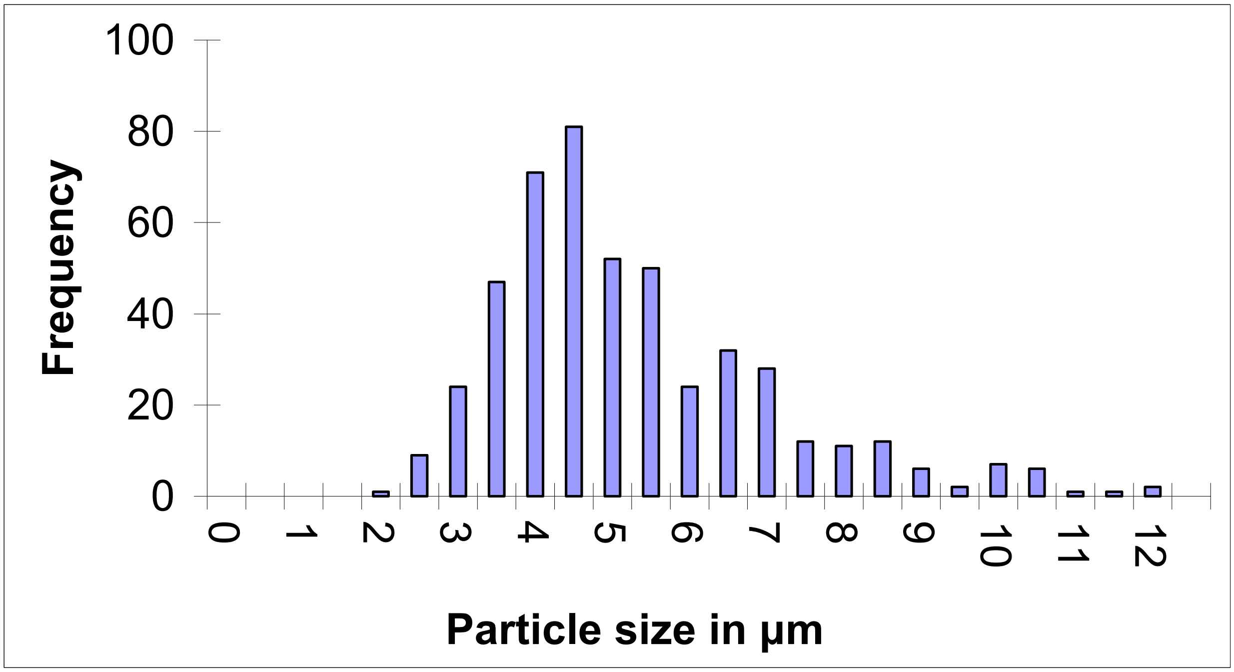

Figure 3. Particle size distribution.

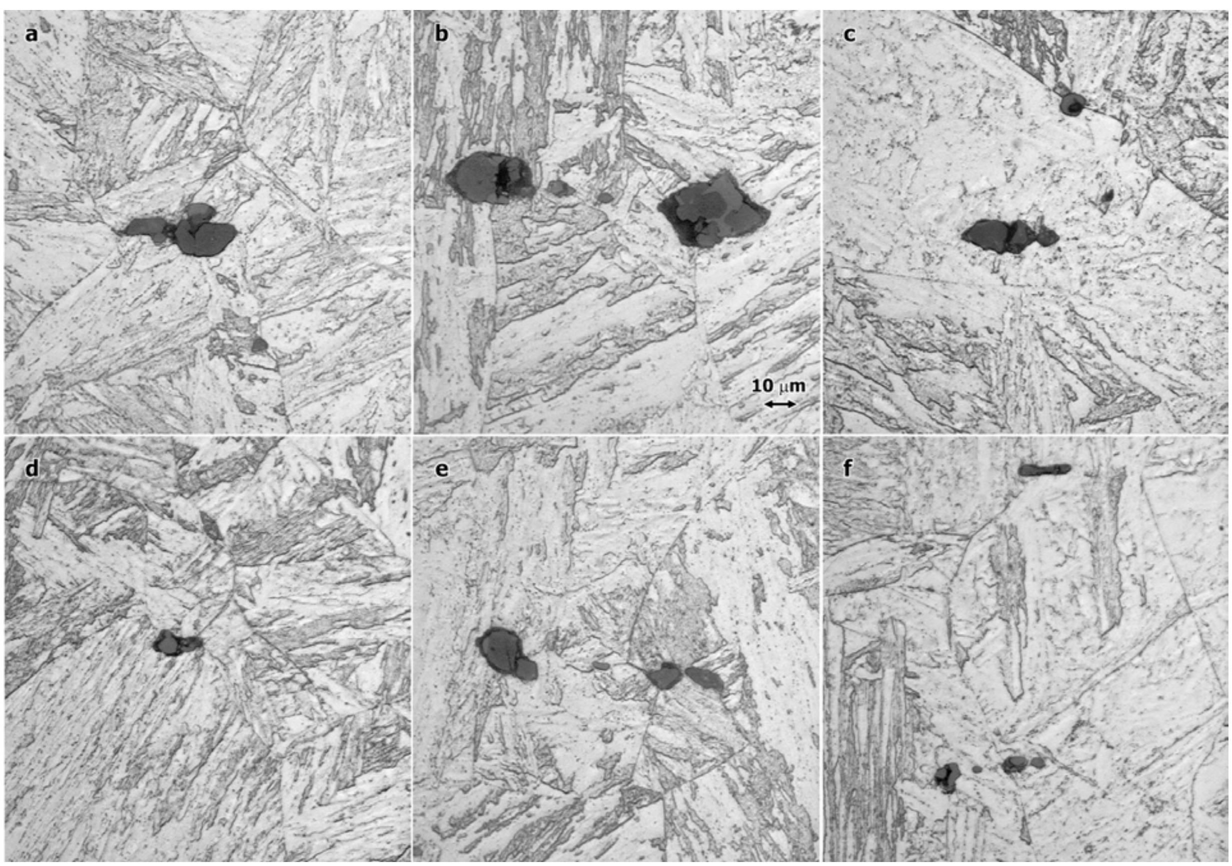

Figure 4. Higher magnification examples of clumped particles.

Scanning Electron Microscopy

The longitudinal section was examined by SEM to determine the chemical nature of the particles. Particles appeared as bright features in backscatter mode, but low magnification images did not contain sufficient resolution to map the distribution of particles. Examples of particles in backscatter mode over a range of magnifications are shown in Figure 5. Note that brightness in backscatter mode indicates higher atomic number. Figure 5c) provides another example of particle clumping. Five particles were selected for compositional analysis and x-ray spectra for two containing the lowest Fe content are provided in Figure 6. The spectra show high Ta and varying levels of Ti. Also, O is at higher levels than C. Therefore, these particles can best be described as tantalum oxide. A best estimate of the composition of the particles is Ta-6.8Cr-10.8Ti-4.0V-4.9Fe-1.7Al (metal content in weight percent) and Ta-9.9C-20.9O-4.7Cr-7.3Ti-2.7V-3.4Fe-0.7Al (total content in weight percent) so Ta levels are on the order of 73% of the metal content (and as high as 79% when Ti is low). As Al is not intentionally added in F82H, it is possible that the Al levels measured arise from alumina contamination, the grinding media.

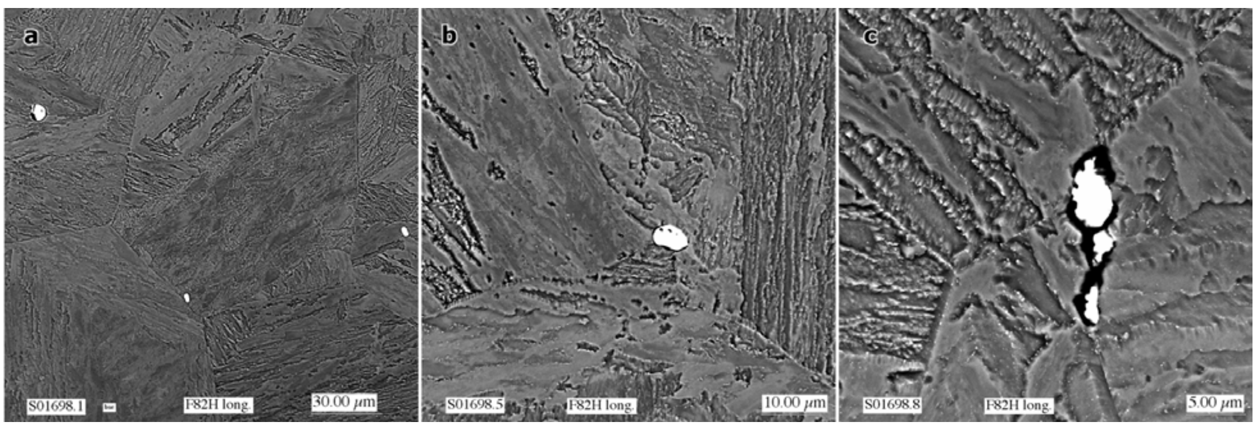

Figure 5. SEM images of particles shown in backscatter mode.

Discussion

After carbide etching, metallographic examination reveals the presence of large tantalum oxide particles in 25 mm plate of F82H produced by JAERI. The particles are approximately 4 μm in diameter but some exceed 12 μm and are slightly elongated in the rolling direction. Note that the 25 mm plate of F82H (heat 5753) contains 0.04 weight percent Ta, in moderate agreement with the estimated volume fraction of 0.1% for the measured tantalum oxide particles [3]. However, particles are uniformly distributed. Therefore, an explanation for reduced fracture toughness cannot be based just on the presence of these large particles. However, clumping of such particles, as appears to be the case particularly in the center of the plate, provides a possible explanation. It can be expected that under plastic strain, undeformable closely spaced particles should easily create insipient cracks between them because insufficient plasticity between particles is available to take up the deformation.

This raises interesting questions about the applicability of Ta additions to martensitic steels in order to promote improved fracture toughness by controlling prior austenite grain size. However, it can be anticipated that clumping of tantalum oxide particles can be reduced by further processing, and perhaps by improvements in melting practice.

Conclusions

Reduced fracture toughness observed in the central portion of 25 mm F82H plate may be attributed to clumped tantalum oxide particles. Therefore, it may be possible to mitigate the problem by elimination of tantalum, or improving melting and/or processes practices.

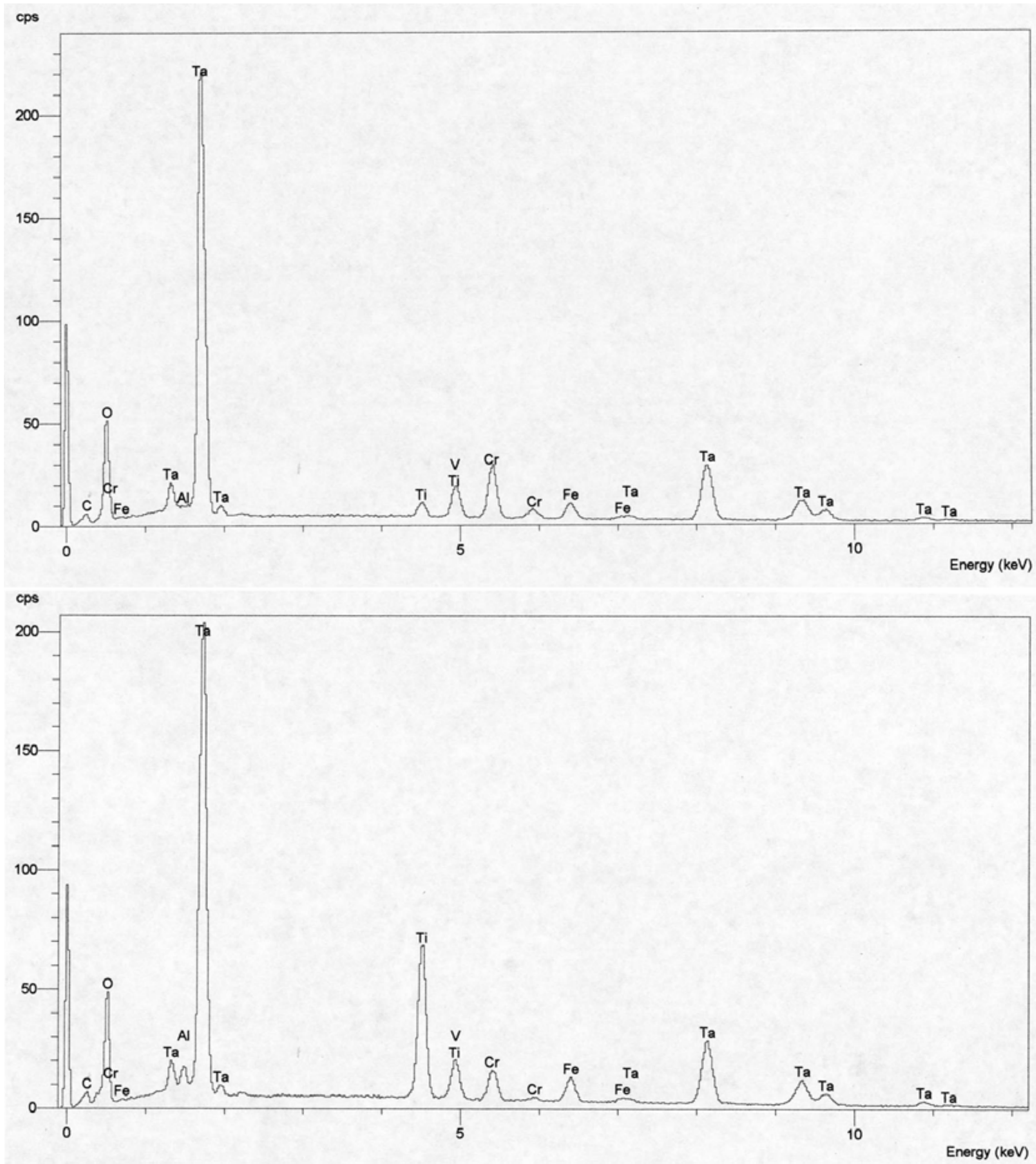

Figure 6. Two examples of x-ray spectra from large particles in 25 mm F82H plate.

Future Work

The effort will be continued as opportunities become available.

References

[1] D. S. Gelles and M. A. Sokolov in DOE/ER-0313/34 (2003).
[2] K. Wallin, A. Laukkanen, and S. Tähtinen in Small Specimen Test Techniques: Fourth Volume, ASTM STP 1418, Eds. M. A. Sokolov, J. D. Landes, and G. E. Lucas (ASTM International, West Conshohocken, PA, 2002) 33.
[3] IEA Summary Report A in the IEA Reduced Activation Ferritic Steel Data Base for F82H compiled by K. Shiba (2003).