

Effect of Heat Treatment on Precipitation in V-5Cr-5Ti heat BL63 - D. S. Gelles and Huaxin Li,
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OBJECTIVE

The objective of this work is to explain changes in mechanical properties of V-5Cr-5Ti heat BL63 due to variations in heat treatment.

SUMMARY

The microstructures of V-5Cr-5Ti heat BL63 are compared following heat treatments at 1125C for 1 h and 1125C for 1 h followed by 890C for 24 h. Following the 890C treatment, precipitate density was increased due to the presence of a moderate density of highly elongated particles. Microchemical analysis showed that these particles often contained both Ti and V, some particles showed minor amounts of Si, S and P, but it was also possible to show that these precipitates were enriched in O rather than C or N. Following the 1125C heat treatment, only Si was found as a minor impurity in large particles, but S could be identified at grain boundaries, which were coated with a fine distribution of precipitates. The embrittlement observed is ascribed to a combination of interstitial solid solution hardening and grain boundary embrittlement, with interstitial hardening likely the dominant factor.

PROGRESS AND STATUS

Introduction

It has been shown that some heats of V-5Cr-5Ti are sensitive to heat treatment variations.¹⁻⁴ The effects can be large. For example, heat treatment of material from Teledyne heat # 832394 (ANL code BL63) at 1125C/1 h (HT1), gives fracture toughness less than 70 kJ/m² when tested at 25C (due to brittle failure), and the ductile to brittle transition temperature (DBTT) as determined by testing 1/3 size Charpy impact specimens is about room temperature.⁴ Properties can be recovered by a treatment of 890C/24 h (HT2).³ The reasons for this behavior are not completely understood. Sulfur concentrations on grain boundaries were found to be higher, by a factor of six, and precipitate concentrations appeared to be decreased for the 1125/1 h treatment.⁴

The purpose of this effort was to examine precipitate structures in BL63 in more detail following HT1 and HT1+HT2 using transmission electron microscopy (TEM) and analytical electron microscopy.

Experimental Procedure

Mechanical properties specimens given HT1 and HT1+HT2, as described in references 3 and 4, were sectioned with a slow speed saw, and sections were punched to provide TEM disks 3 mm in diameter by 0.25 mm thick. Samples were thinned in a commercial twin jet polishing apparatus using a solution of 5% sulfuric acid in methanol operating at 40 V and -20C with a moderate pump speed.

Specimens were examined both in a JEOL 1200EX electron microscope operating at 120 KeV (to provide microstructural information) and a JEOL 2010F electron microscope operating at 200 KeV (to

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provide microchemical information). Precipitates were attacked more slowly than the grain matrix during electropolishing, making it possible to restrict precipitate composition spectra to particles hanging over the edge of the thin foil, reducing the effect of the surrounding matrix. Beam conditions on the 2010F provide an electron probe approximately 1 nm in diameter for compositional analysis.

Results

Precipitate Distributions

The microstructures of the two specimen conditions revealed differences in precipitate structures. HT1 produced generally clean microstructures, with a very low density of large precipitates, often associated with grain boundaries and triple points. However, unusual contrast was observed on some grain boundaries, indicative of fine precipitation. The HT1+HT2 condition contained a higher density of precipitate particles, generally decorating grain boundaries, but also in groups in the matrix. Particles in the matrix were generally rod shaped, but at grain boundaries they were more globular forming complex particle arrays. As precipitation is generally coarser following heat treatment at higher temperatures, it is possible that the fine precipitate found at grain boundaries following HT1 had formed during slow furnace cooling.

Examples of these structures are provided in Figure 1. Figures 1a and b provide low magnification examples of grain boundary structures in V-5Cr-5Ti heat BL63 following heat treatment at 1125C for 1 h and furnace cooled (HT1). Figure 1c shows particles at a grain boundary in a specimen of the same material heat treated for a further 24 h at 890C and then furnace cooled (HT1+HT2). Figures 1d, e, and f give examples of precipitate structures at higher magnification, showing the fine grain boundary structure following HT1 in Figure 1d, the coarser grain boundary structure following HT1+HT2 in Figure 1e and precipitation in the matrix following HT1+HT2 in Figure 1f.

Therefore, HT1+HT2 provides a microstructure with a higher density of second phase particles, probably at a higher volume fraction. However, the precipitate density actually on grain boundaries may be similar for the two conditions because a fine distribution of particles is found on boundaries following HT1.

Precipitate Compositions

Compositional spectra were obtained for precipitates extending over the edge of foils prepared with large perforations, to provide information on a large number of particles. Approximately eight precipitate particles were analyzed for condition HT1 and 30 for HT1+HT2. In addition, seven grain boundary areas were analyzed in condition HT1. In both cases, particles were found for each of three composition ranges, 1) high Ti with V, 2) approximately equal levels of Ti and V, and 3) high V with Ti. In both conditions, the high Ti case (1) was the most frequently observed. The statistical details are provided in Table 1. However, significant differences could be identified between the two conditions with regard to S, P, Si and O. Also of note were two flakes of material, found on the HT1 condition, that showed unusually high S content.

Table 1. Number of Precipitate Particles Examined with Observed Minor Elements noted

Treatment	# V rich	# (V,Ti)	# Ti rich
HT1	2 showing low Si, S	1 showing low Si, S	5 showing Si
HT1+HT2	9 showing O, C, Si, P, and S	5 showing C, O	17 showing O, Si, P, and S

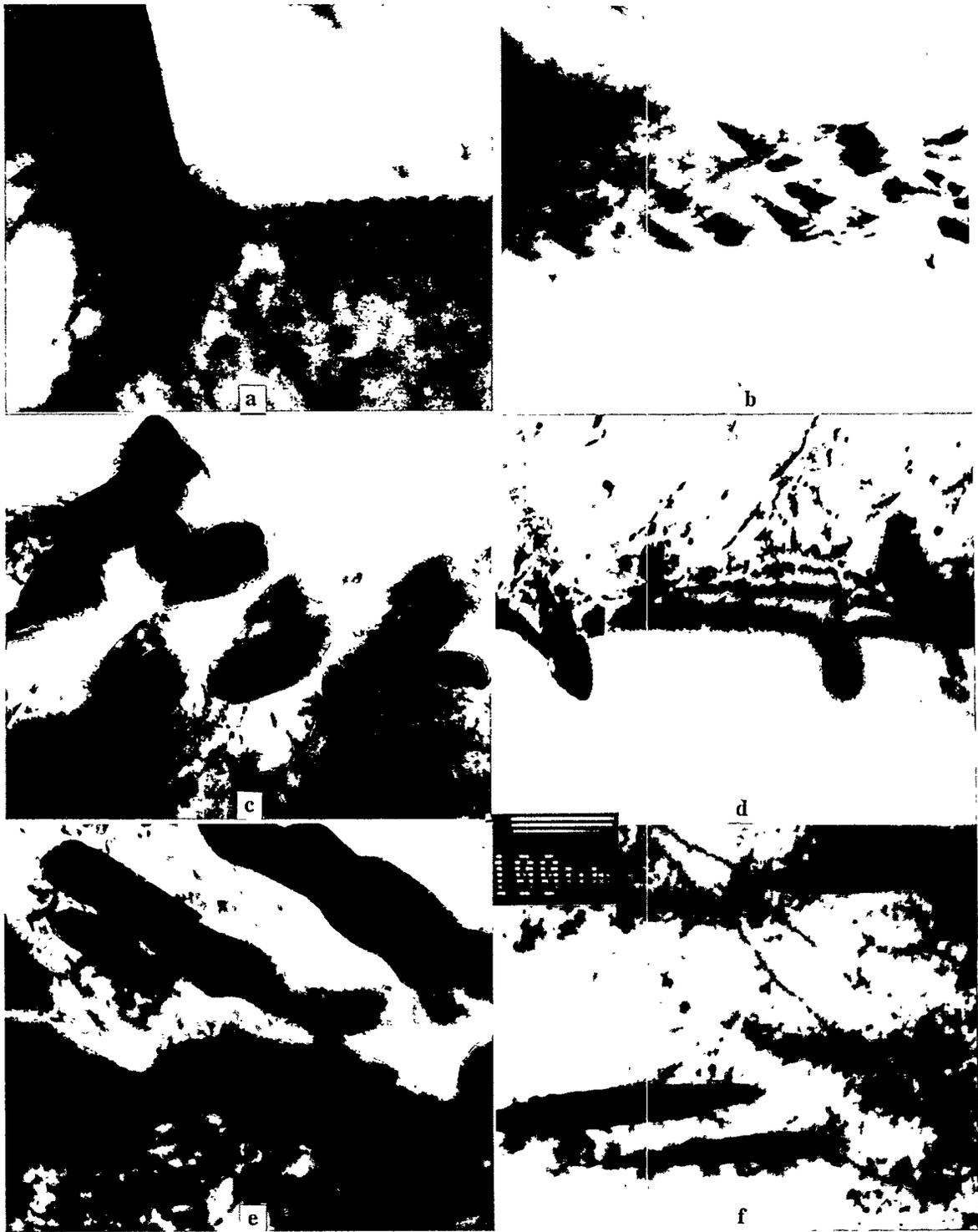


Figure 1. Precipitate structures in V-5Cr-5Ti HT1, (a), (b), and (d), and in HT1+HT2, (c), (e), and (f).

Examples of x-ray spectra for representative particles from each condition are provided in Figure 2 along with a matrix spectrum for the HT1+HT2 condition. Figures 2 a through d are from the HT1 condition and 2 e through h are from the HT1+HT2 condition. Figure 2h, showing a typical matrix spectrum, should be examined first. It displays peaks typical of a material high in V, with low levels of Ti and Cr, but the V-K_β peak overlaps the Cr-K_α. The V-L and Ti-L peaks can be identified in the low energy range at 0.5 and 0.25 KeV, respectively. These L peaks provide a basis for comparison with light elements C, N, and O, which overlap this region, in effect providing information on the efficiency of the x-ray detecting system for light elements. Figure 2a shows the spectrum for a particle that contained high V levels in condition HT1. A strong C peak is indicated, but it should be noted that this peak grew relative to the other peaks during acquisition, indicating that it was largely due to C contamination, and therefore conclusions regarding C must be taken with care. Figures 2b and c show particles containing approximately equal levels of V and Ti, and high Ti, respectively. Of particular note is the fact that the peak at 0.5 KeV is larger in both cases than that in Figure 2h, indicating the presence of O. Figure 2d shows an example of a spectrum taken of a grain boundary region, which indicates the presence of S, by the peak at 2.3 KeV.

Figures 2e, 2f, and 2g show similar features for precipitates with high V, with approximately equal levels of V and Ti, and with high Ti, respectively. Three features are of particular note. The C contamination was not a problem with this specimen, so C peaks are often absent. Oxygen is indicated in all precipitates, and the O levels are significantly higher than in the HT1 spectra. Also, evidence of Si, P, and S can be found in several particles, particularly in the case of Figure 2g for the high Ti containing particle where a strong S peak can be seen at 2.3 KeV.

These results demonstrate that heat treatment at 890°C for 24 h results in higher O, S, P, and perhaps Si levels in precipitate particles, compared to the situation following heat treatment at 1125°C for 1 h. The concentration of these elements left in solid solution would therefore be significantly higher following the high temperature treatment. The results also confirm that S is found in grain boundary regions following the high temperature heat treatment, and this S may be in the form of thin layers, as indicated by the flake material identified with high sulfur.

Discussion

The present work provides two possible explanations for the observed embrittlement and reduction of toughness arising from heat treatment of V-5Cr-5Ti heat BL63 at 1125°C for 1 h: interstitial hardening and grain boundary embrittlement during furnace cooling.

We find that recovery of ductility and toughness properties is achieved by concurrent precipitation of (Ti,V)O. This removes not only Ti from solid solution, but also significant amounts of O. Precipitation would generally increase strength and reduce ductility, but the particle spacing is quite large, so the major effect is expected to occur from interstitial hardening or lack thereof. Precipitates also incorporate significant amounts of P and S, apparently reducing the concentrations in solid solution and at grain boundaries. Therefore, precipitation is expected to not only reduce interstitial hardening, but also the level of S at grain boundaries.

We also find that precipitation does develop on grain boundaries following the 1125°C treatment. The precipitate is at high density and in the form of fine particles directly associated with the boundary. As noted, it is expected that such precipitation formed on cooling rather than at temperature, in part because a much coarser structure is expected at higher temperature, but also because it is shown that a strong tendency exists to form such structures at somewhat lower temperatures, and a slow cooling process such as furnace cooling would allow such precipitates to form, but to a less extent than for a long time at temperature.

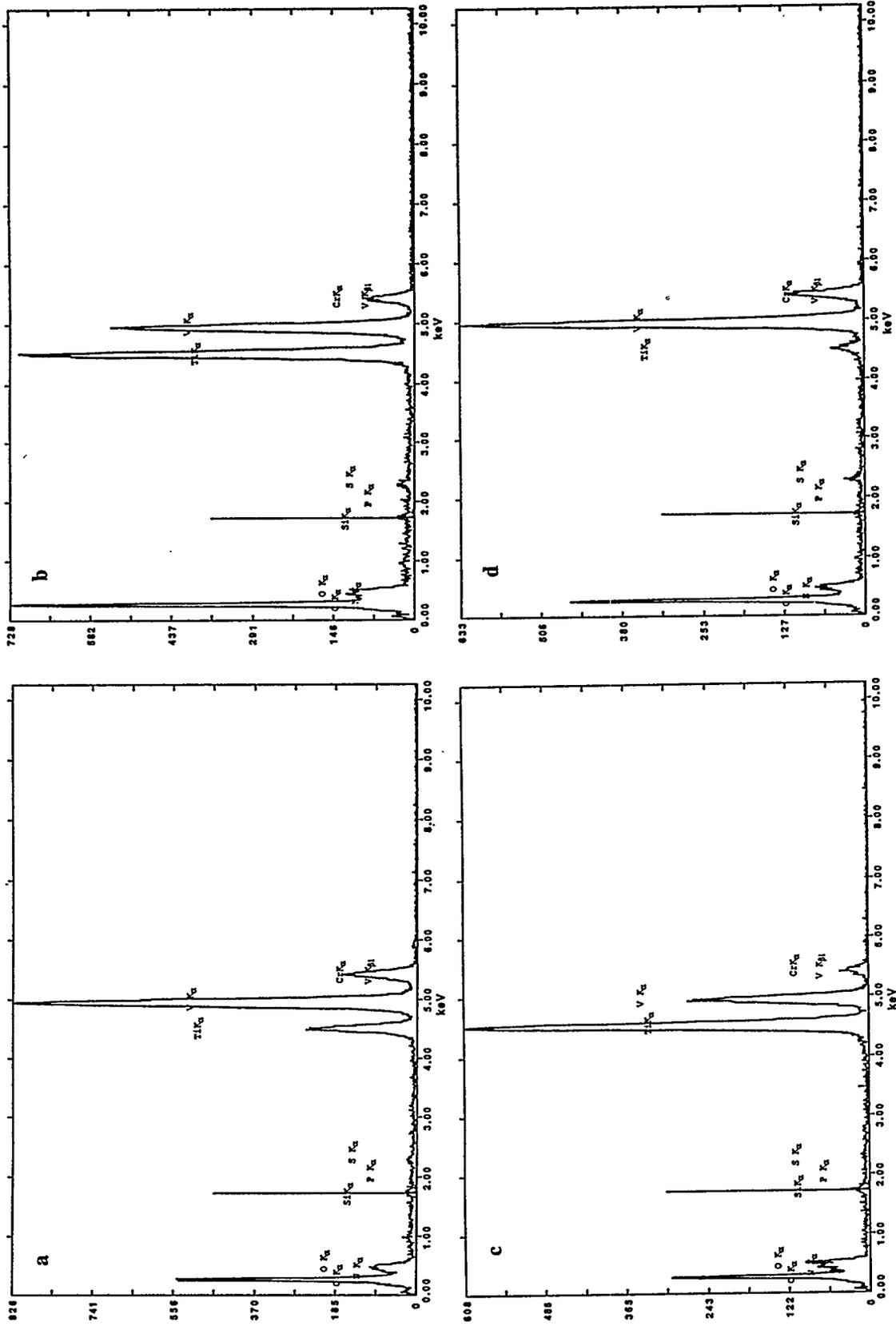


Figure 2. X-ray Spectra for a specimen with HTI in (a), (b), (c), and (d). Continued on Next Page.

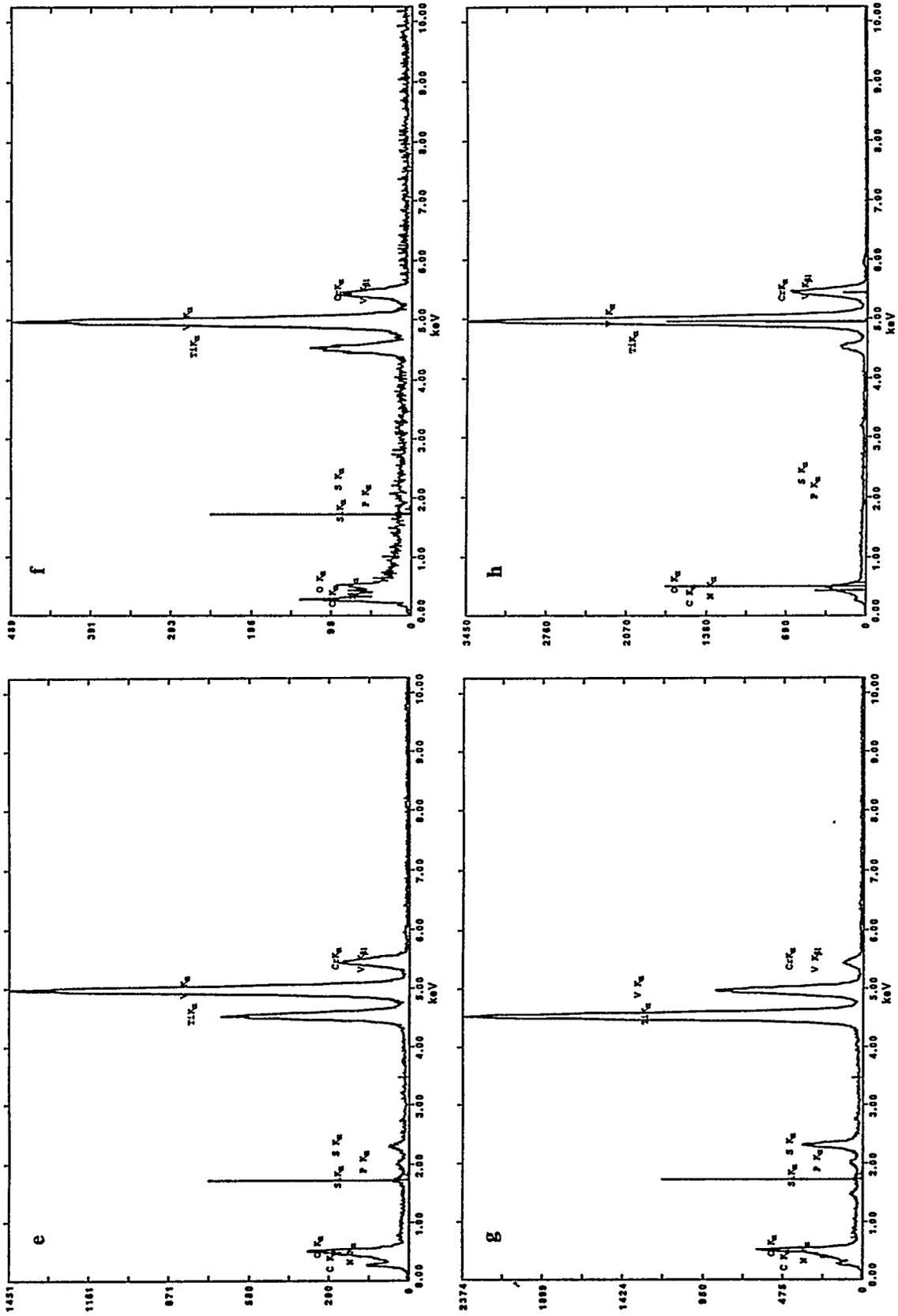


Figure 2. Continued Showing X-ray Spectra for a specimen with HT1+HT2 in (e), (f), (g), and (h).

The question can then be asked, which of these processes is more important? It can be noted that failure as a result of embrittlement consisted of both brittle transgranular cleavage and failure at grain boundaries. Therefore, both grain boundary embrittlement and hardening play a role because the failure mode changed from dimple rupture to transgranular cleavage. As interstitial hardening can explain both cleavage and intergranular features whereas grain boundary embrittlement cannot, it is anticipated that precipitation of (Ti,V)O is the more important process because it reduces the levels of interstitial O available for solid solution hardening.

CONCLUSIONS

The microstructures of two conditions of V-5Cr-5Ti heat BL63 have been compared to explain the embrittlement arising from heat treatment at 1125°C for 1 h (HT1) and the recovery of ductility and toughness from heat treatment at 890°C for 24 h (HT1+HT2). Differences in precipitation are found between the two conditions such that more precipitation, consisting of (Ti,V)O and containing Si, P and S, develops at 890°C and a high density of fine precipitates is found on grain boundaries following heat treatment at 1125°C. This grain boundary precipitation is believed to develop during furnace cooling. Enhanced sulfur concentrations are confirmed following the 1125°C heat treatment. The embrittlement is ascribed to a combination of interstitial solid solution hardening and grain boundary embrittlement, with interstitial hardening likely the dominant factor.

FUTURE WORK

This effort will continue as further specimens are available.

REFERENCES

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