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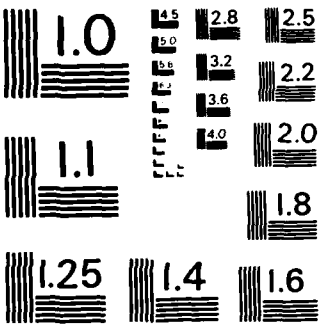

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A STUDY OF PRECIPITATION AND SEGREGATION ON AN ATOMIC SCALE OF 11at.%Cr-1at.%Mo HEAT RESISTING STEEL BY ATOM-PROBE AND TEM

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An 11at.%Cr-1at.% Mo commercial steel which contains C, Si, Mn, Nb and V as minor elements was investigated by atom-probe (AP) and transmission electron microscopy (TEM). This steel has an excellent heat resisting property at 600 - 650°C but undergoes the ductile - brittle transition in this temperature range. The following results were obtained: (1) After solution treatment the FIM image was homogeneous on an atomic scale. (2) After tempering at 650°C for 1 hr. a precipitate was detected AP analysis showed that the precipitate was M(C+N) where M is Nb, Cr, V, Mo and Fe. (3) After tempering at 700°C for 1 hr., a

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precipitate was a Fe/Cr ratio of 46/42 (sigma phase) was detected.
(4) After tempering at 700°C for 1 hr. a precipitate Fe/CR ratio of 60/40 (sigma phase) was also observed at the lath boundary, and adjacent to this region the chromium concentration decreased to 1%.

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Abstract - An 11at.%Cr-1at.%Mo commercial steel which contains C, Si, Mn, Nb and V as minor elements was investigated by atom-probe (AP) and transmission electron microscopy (TEM). This steel has an excellent heat resisting property at 600 - 650°C but undergoes the ductile - brittle transition in this temperature range. The following results were obtained: (1) After solution treatment the FIM image was homogeneous on an atomic scale. (2) After tempering at 650°C for 1hr a precipitate was detected. AP analysis showed that the precipitate was M(C+N) where M is Nb, Cr, V, Mo and Fe. (3) After tempering at 700°C for 1hr, a precipitate with a Fe/Cr ratio of 46/42 (sigma phase) was detected. (4) After tempering at 700°C for 1hr a precipitate Fe/Cr ratio of 60/40 (sigma phase) was also observed at the lath boundary, and adjacent to this region the chromium concentration decreased to 1%.

I - INTRODUCTION

In the research and development of alloy steels, the problems of micro-precipitation and microsegregation are becoming more important for control of the properties of steels. Hitherto, these studies have been carried out by auger electron spectroscopy /1,2/. However this method is not good enough to know the microstructure on an atomic scale. Therefore, it is meaningful to use a field ion microscope (FIM) and AP to solve these problems. There have been many reports on steel /3,4,5/, but in this study, a 11at.%Cr-1at.%Mo commercial steel, which includes C, Si, Mn, Nb and V as minor elements, was observed. This steel has an excellent heat resisting property at 600 - 650°C but there is the problem of the ductile - brittle transition in this temperature range /6/. Because of this phenomenon it is very important to study and clarify the microstructures of this steel. This steel is conventionally used after quenching and tempering and is known to have microstructures of dual phase with tempered martensite and sigma ferrite.

II - EXPERIMENTAL PROCEDURE

The bulk chemical composition of the heat resisting steel used in this investigation is given in Table 1. This material was melted in air, and after rolling and annealing, finished with wire drawing into 0.2 mm wire. The solution treatment was carried out at 1050°C for 30min in a vacuum of $2x10^{-3}$Pa. Then, tempering was performed at 650°C and 750°C for 1hr, respectively. As for FIM imaging, Ne was used at $2x10^{-4}$Pa, and the observation was performed at 30K. For TEM observation, a JEOL 100B was used.

Table 1 - Chemical composition.

	C	Cr	Si	Mn	P	S	Mo	V	Nb	Fe
wt.%	0.05	10.0	0.52	0.56	0.009	0.007	2.0	0.10	0.05	bal.
at.%	0.26	10.8	1.04	0.57	0.016	0.012	1.2	0.11	0.03	bal.

III - EXPERIMENTAL RESULTS AND DISCUSSION

1 - MC type microprecipitate

Photo 1 shows the FIM microphotograph of a solution treated specimen. After solution treatment the (110) surface (in the center) was observed and the bright spots were uniformly observed. In this case, the chemical composition measured by AP was the same as that of the chemical analysis. After tempering at 650°C for 1hr microprecipitates were observed in both the FIM image and the TEM image, which are shown in Photo 2(a) and (b), respectively.

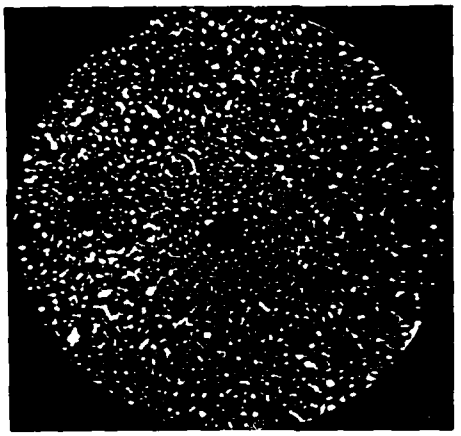
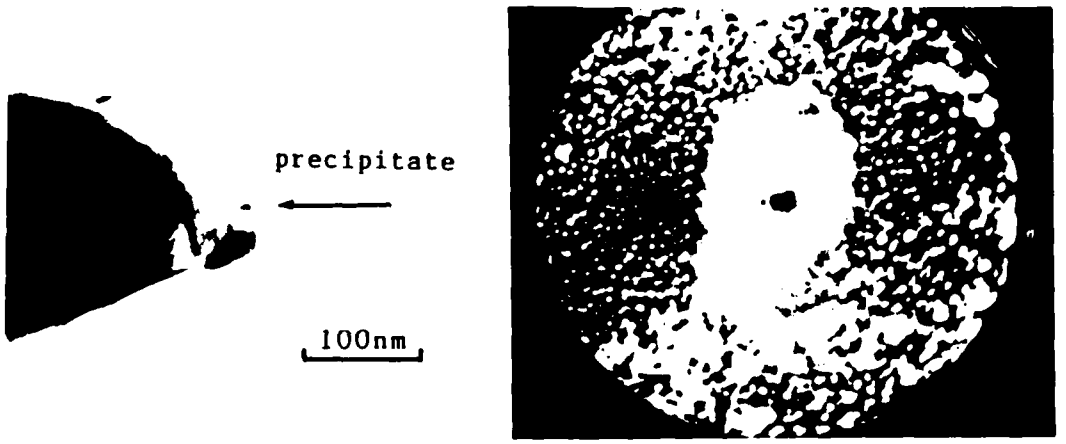


Photo 1 - Field ion microphotograph of the heat resisting steel solution annealed at 1050°C for 30 min.



(a) (b)

Photo 2 - TEM image and FIM image of the heat resisting steel tempered at 650°C for 1hr. (a) TEM image and (b) FIM image of the same specimen.

As shown in Photo 2(b) the shape of the pr cipitate was elliptical and the size was about 20x10nm^2. Fig. 1 shows the results of an AP analysis of this precipitate. A total of 3200 atoms were analysed. Since the probe-hole limits the number of atoms per atomic plane to about 25 substitutional atoms, the thickness of the precipitate was found to be 14 substitutional atomic layers with 5 substitutional atomic transient layers including the phase boundary. The AP analysis showed that the precipitate consisted of 64Nb, 18Cr, 18V, 6Mo and 3Fe substitutional atoms with 30C and 70N interstitial atoms. Therefore, this precipitate was revealed to be M(C+N) mainly consisting of NbN, because the total metal atoms was 109 and the total C or N atoms was 100. In the case of the matrix, the AP analysis showed uniformity with 227OFe, 297Cr, 29Mo, 18Mn, 3V and 23 N atoms. In the transition layers there were no significant anomalies. Table 2 shows the concentrations of each element in the precipitate, matrix and transient layer including the phase boundary.

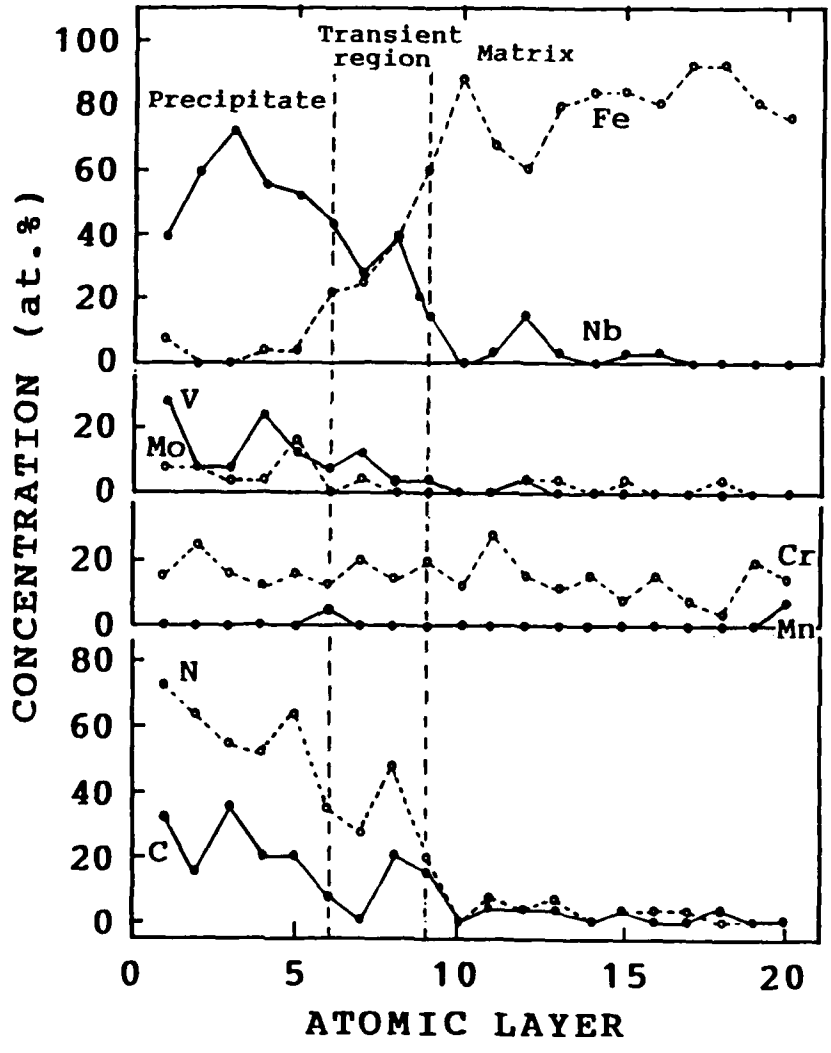


Fig. 1 - Depth profile of the chemical composition in the precipitate, matrix and transient region. The thickness of the precipitate was revealed to be 14 atomic layers with 5 atomic transient layers.

Table 2 - Concentration of the precipitate, matrix and transient region.

	Fe	Cr	Nb	Mo	Mn	V	C	N
Precipitate	1.4	8.6	30.6	2.9	0	8.6	14.4	33.5
Transient region	42.1	12.9	15.7	0	0	3.6	6.4	19.3
Matrix	86.4	11.3	0	1.1	0.7	0.1	0	0.9

2 - Sigma phase microprecipitate

2 - 1 Precipitate along grain boundary

Photo 3 shows the microprecipitate along the grain boundary. The AP analyses are shownin Fig. 2 and Fig. 3. Fig. 2 shows that the precipitate is mainly Fe and Cr atoms with a ratio of 46/42 (sigma phase) Fig. 3 shows that minor metallic elements were also detected. Usually the sigma phase can be observed only when the Cr concentration is above 17 at.% in iron chromium alloy. But in this study the average concentration of chromium was 11at.%. It is , however, considered that near the grain boundary, the free energy increases with strain energy and there is the possibility to find the sigma phase as the "local equilibrium state" even thoughthe chrominium concentration is lower then 17 at.%. In this case the concentration of Cr of the surrounding matrix showed some deficiency at about 6.9 at.%, and Fe was 91.8%. At the interface of the precipitate, the concentration of Cr was found to be the averaged value between the precipitate and surrounding matrix.

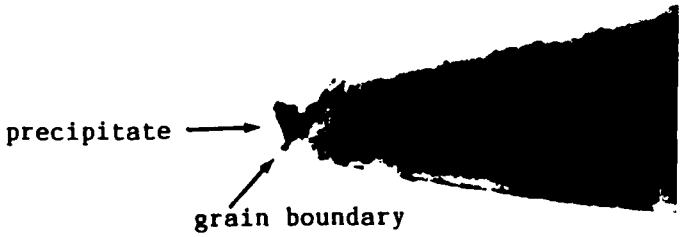


Photo 3 - Sigma phase microprecipitate along the grain boundary.

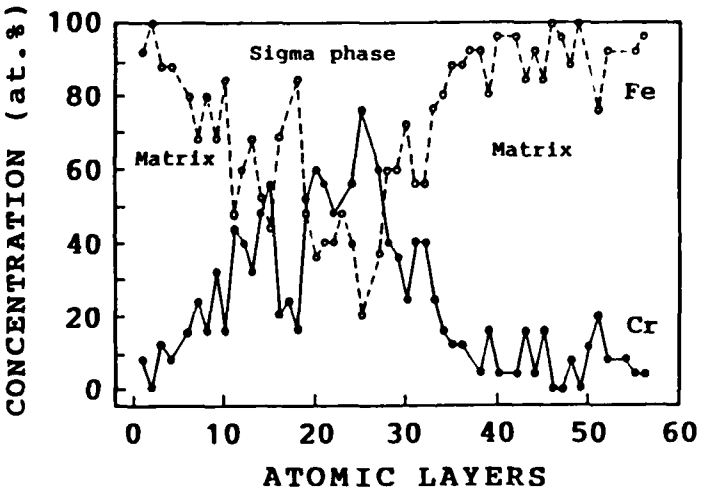


Fig. 2 - Concentration of Fe and Cr atoms per atomic layer for the heat resisting steel tempered at 700°C for 1hr.

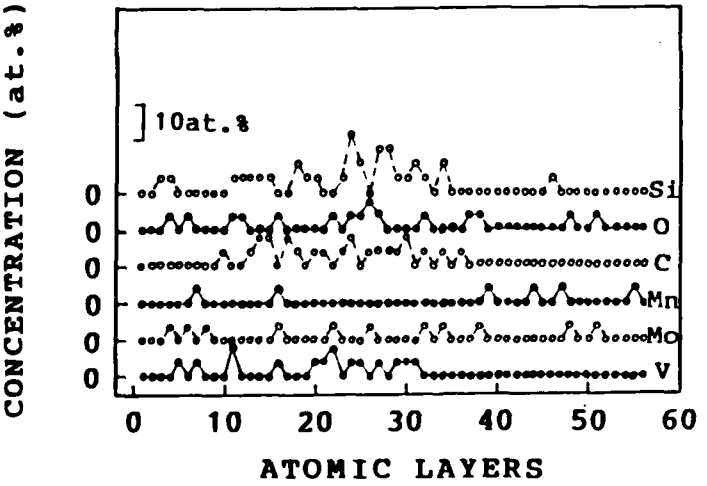


Fig. 3 - Concentration of Si, O, C, Mo, V and Mn atoms per atomic layer for heat resisting steel tempered at 700°C for 1hr.

2 - 2 Precipitate along the lath boundary

Photo 4 shows a TEM image of the tip specimen tempered at 700°C for 1hr. Photo 5 shows the FIM image after evaporation of the surface layer of the tip shown in Photo 4. By evaporating the tip, the AP analysis was performed across the lath boundary. From FIM and AP analysis the lath boundary in tempered state seems to be a small angle boundary including both tilt and twist. Fig. 4 shows the depth profile of chemical composition. In the precipitate the ratio of Fe/Cr was 60/40 (sigma phase). This figure shows that there is enrichment of chromium along the grain boundary and depletion of chromium outside this region.

Photo 4 - TEM image of the tip specimen tempered at 700°C for 1hr.

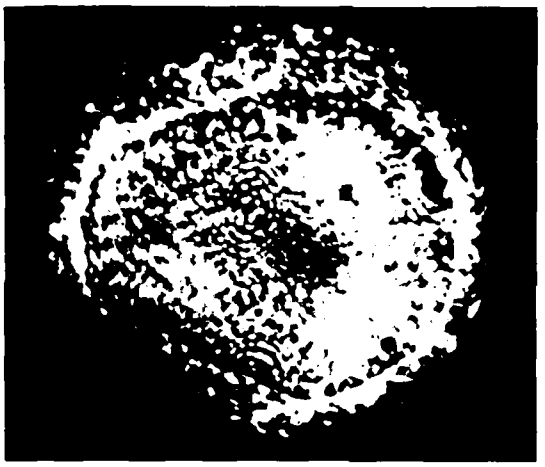


Photo 5 - FIM image after evaporation of the surface layer of the tip shown in Photo 4.

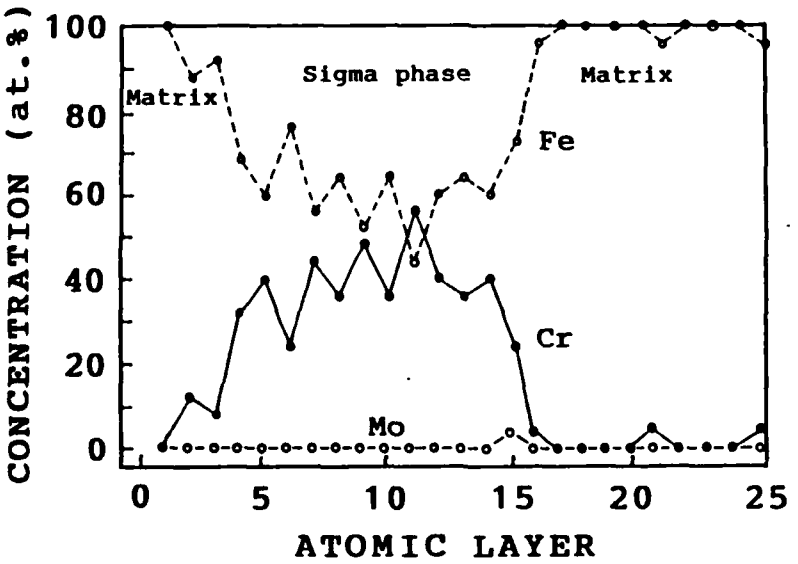


Fig. 4 - Concentration of Fe, Cr and Mn atoms per atomic layer for the heat resisting steel tempered at 700°C for 1hr.

IV - SUMMARY

In this study the microprecipitate and microsegregation in an 1lat.%Cr-lat.%Mo heat resisting steel were revealed.

(1) After solution treatment the FIM image showed homogeneity and no precipitate was observed.

(2) After tempering at 650°C for 1hr precipitates of about $20x10nm^2$ were detected by both TEM and FIM. From the results of the AP measurements, the precipitate was revealed to be M(C+N) where M is 64Nb, 18Cr, 18V, 6Mo and 3Fe atoms (total 109 atoms), and C is 30 and N is 70 atoms (total 100 atoms). There was no segregation to the transient region of the phase boundary.

(3) After tempering at 700°C for 1hr, the precipitate, which has the high chromium concentration, was detected along the grain boundary. In the precipitate the ratio of Fe/Cr was 46/42 (sigma phase) with additional minor elements. There was no significant segregation at the interface between the precipitate and matrix.

(4) After tempering at 700°C for 1hr, a high chromium concentration was observed at the lath boundary. Outside this layer the chromium concentration decreased to 1%. In the precipitate the ratio of Fe/Cr was 60/40 (sigma phase).

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