Type of presentation: Oral

MS-4-O-3131 Transmission Electron Microscopy analysis of sigma (σ)-phase formation mechanisms in high chromium content iron-chromium model alloys with small addition of molybdenum for high temperature application

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One of the well-known intermetallic phases which form in the Fe-Cr system is the sigma (σ)-phase. It has a tetragonal unit cell (space group P_{42}/MNM) with a = 8.80 Å and c = 4.54 Å [1]. In general, the σ-phase forms during long-time exposure in the temperature range of 565 to 980°C and it's composition varies quite widely which makes it difficult to give an exact stoichiometry. The formation of the σ-phase is one of the main reasons for the deterioration of stainless steel properties. As an example, it has been reported that the precipitation of σ-phase on grain boundaries consumes chromium and as a result, there is a loss in corrosion resistance [2]. The present investigation was conducted to investigate the σ-phase formation caused by the addition of molybdenum as one of the ferrite stabilizing alloy elements in order to control the σ-phase nucleation mechanisms.
A high purity model alloy containing 68 wt% Fe, 30 wt% Cr and 2 wt% Mo, manufactured by ThyssenKruppVDM, was annealed in vacuum for 1000 h at 700°C. Later, the specimen was prepared using the focused ion beam (FIB) technique in a FIB Strata 205 from FEI. Microstructural analyses were carried out using the Libra 200 FE and Titan-T TEMs under an acceleration voltage of 200 KV and 300 KV respectively.
The formation of the σ-phase along the grain boundaries as well as within the grains are shown in Figure 1. Two types of precipitates with contrast difference are detected on σ-phases formed on grain boundaries (figure 1.b). Figure 2 shows the bright-field (BF) TEM images and SAED patterns of these two precipitates. The BF images, figure 2.a and 2.c, show that the bright precipitate (marked as L2 in fig 1.b) contains a higher density of planar faults than the dark one (marked as L1 on fig 1.b). Furthermore, the analyses of the SAED patterns also confirms that the dark and bright precipitates were identified as chromium-carbide and chi (χ)-phase, respectively (fig 2.c and 2.f). The BF images also revealed that part of the σ-phase close to the chromium-carbide precipitates has less planar faults than the one close to χ-phase precipitates.
The matrix/σ-phase interface from σ-phase formed on grain boundaries and inner grains (marked L1 and L3 in figure 1 respectively) were further investigated by means of high-resolution imaging technique. Figure 3 shows an example of the high-resolution (HR) image of σ-phase formed on grain boundaries which contains faults at the interface.

References

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[2] N. Lopez, M. Cid, and M. Puiggali, Influence of σ-phase on mechanical properties and corrosion resistance of duplex stainless steels, Corr. Sci, vol. 41, no. 8, pp. 1615-1631, 1999.

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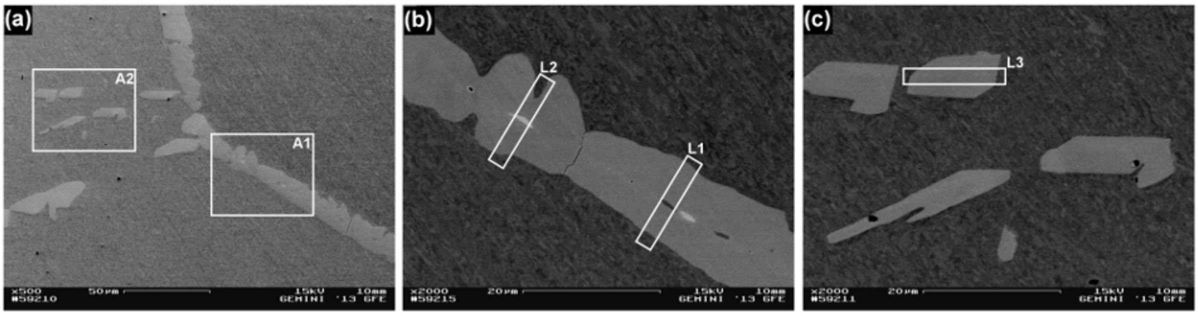

Fig. 1: (a) SEM-BSE micrographs of the investigated specimens showing σ-phase precipitated on grain boundaries and inside grains as well as selected areas marked by A1 (b) and A2 (c) where FIB lamella were prepared.

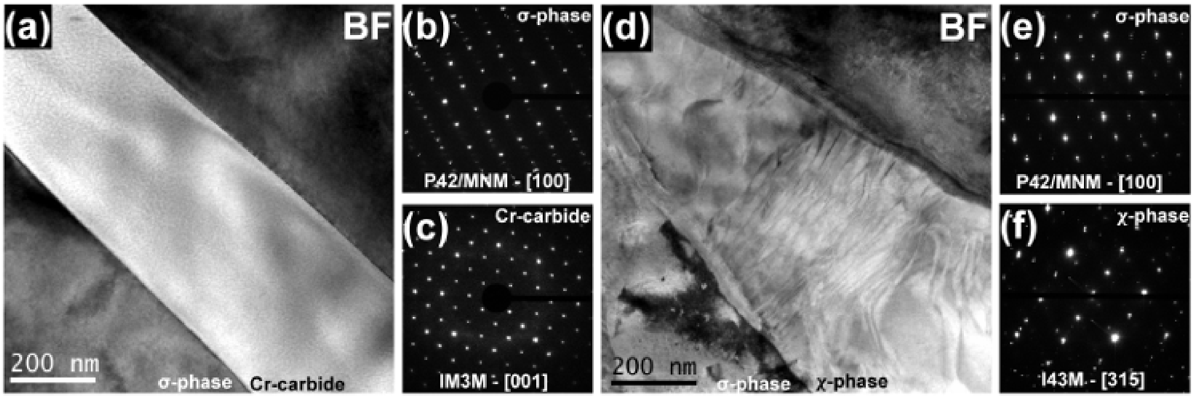

Fig. 2: Bright-field (BF) images and SAED patterns of two precipitates, Cr-carbides and χ-phase, which were found on σ-phase formed on grain boundaries (marked by L1 and L2 on fig 1.b). The BF and SAED pattern show that the σ-phase contains numerous planar faults.

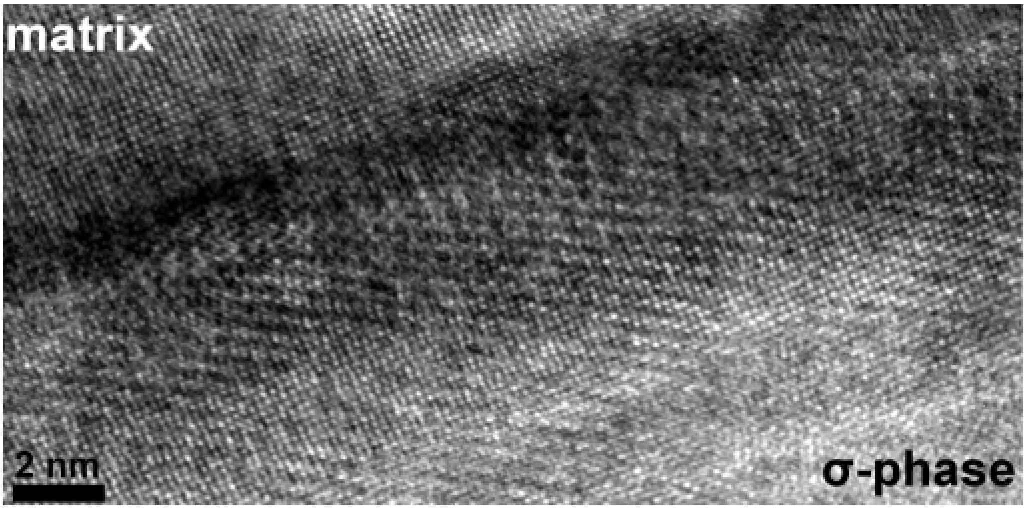

Fig. 3: High-resolution (HR) image taken from matrix/σ-phase interface of σ-phase formed on grain boundary (marked L1 in figure 1.b) with the electron beam parallel to the [001] matrix zone axis.