Z-phase is a complex nitride with CrNbN stoichiometry and tetragonal crystal lattice [1]. It was first found in 1950s in austenitic steels and its precipitates have been attributed beneficial strengthening effects. A renewed interest in Z-phase launched in 1990s when it was detected in 9-12% Cr creep resistant ferritic steels for power plant applications [2-3]. Slowly precipitating Z-phase in ferritic steels consumes fine dispersion of (Nb,V)(C,N) particles and severely deteriorates long term creep properties. A precise knowledge of thermodynamic parameters of Z-phase is crucial for complete assessment of multicomponent systems and for reliable prediction of creep life.

In this work we study the Z-phase in model Cr-Nb-N alloys by means of analytical electron microscopy (SEM and TEM). Cr-Nb alloys with 1-14 at.% Nb were arc melted under the Ar+N\textsubscript{2} atmosphere. The resulting content of N was 15-20 at.%. Samples were annealed for a long time at 1100 and 1300 °C to reach states close to thermodynamic equilibrium. Here we present the results obtained on 70Cr-14Nb-16N (at.%) sample annealed at 1100 °C for 48 days, i.e. the one after the longest annealing time where the Z-phase was found. A TESCAN LYRA 3XMU FEG/SEM×FIB scanning electron microscope and a Philips CM12 STEM transmission electron microscope (both equipped with an XMax 80 Oxford Instruments detector for energy dispersive X-ray (EDX) analyses) were used for microstructural studies.

Figure 1a shows microstructure with a distribution of bright coarse needles/plates and with a mixture of two phases between needles. A closer look (Fig. 1b) reveals fine two-phase microstructure also inside needles. This indicates the dissolution of (Cr,Nb)\textsubscript{2}N phase originally present in as cast alloy and its substitution by Z-phase and Cr. The stable equilibrium of the reported sample at 1100 °C is presumably Cr solid solution + Z-phase. X-ray analysis confirmed the Z-phase together with a bcc Cr phase and traces of (Cr,Nb)\textsubscript{2}N. EDX analyses in SEM support the conclusion that there are only two equilibrium phases; one of them (the dark regions in SEM micrographs) around 98Cr-1Nb-1N (at.%) and the second one close to 1:1:1 stoichiometry. N content in both phases was confirmed also by wave dispersion X-ray analyses. Elemental distribution is clearly shown on EDX maps in Figure 2. Identification of phases was accomplished by TEM inspection of thin lamella prepared by focused ion beam (FIB) in SEM (Figures 1c and 3).


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Fig. 1: SEM micrographs of Cr-Nb-N sample, signal of backscattered electrons. Low magnification overview (a), a detail of needle microstructure (b) and a scene from TEM lamella preparation (c).

Fig. 2: A detail of microstructure (SEM, signal of backscattered electrons) with elemental distribution maps measured by EDX.

Fig. 3: TEM micrographs of lamella prepared by FIB. The whole lamella at low magnification (a), a detail of two-phase microstructure (b) and SAD patterns of the two phases – Cr solid solution and Z-phase (c).