Experimental report

18/08/2017

Proposal: 1-01-150 **Council:** 4/2016

Title: In-situ SANS study on influence ofboron addition on TaC precipitate stability in Co-Re alloys at high temperatures

Research area: Materials

This proposal is a new proposal

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Experimental team: Lukas KARGE

Local contacts: Dirk HONECKER

Samples: Co80Re17Ta1.2C1.08B0.1#2
Co80Re17Ta1.2C1.08B0.4#2
Co80Re17Ta1.2C1.08B0.1

Instrument	Requested days	Allocated days	From	To
D22	4	0		
D33	4	4	12/09/2016	16/09/2016

Abstract:

Co-Re alloys are being developed to supplement Ni-base superalloys in future gas turbines for ultra-high temperature applications. Previous small-angle neutron scattering experiments at FRM II confirmed stability of fine (3-100 nm) Tantalum Carbides (TaC) up to 1200 °C. These precipitates are promising for strengthening the alloy at high temperatures. Addition of Boron increases strength and ductility in the alloy by making the grain boundaries more cohesive. However, it is known that B interacts with carbides in steels and Ni-alloys and hence might have an influence on stability of fine TaC. Neutron diffraction found an allotropic phase transformation in the Co-solid solution matrix in Co-Re alloys with varying C/Ta ratio at 1000 – 1300 °C, affecting stability of fine TaC. In a German Science Foundation (DFG) funded project jointly between TU Muenchen and TU-Braunschweig the main aim is to optimize the alloy composition with respect to the C/Ta ratio and boron content. In-situ SANS experiment at high temperatures is the method of choice to investigate the formation, evolution and stability of the fine TaC precipitates in alloys containing 0.1 to 0.4 at. % B.

In-situ SANS study on influence ofboron addition on TaC precipitate stability in Co-Re alloys at high temperatures

Experimental team:

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Instrument: D33

Local contact: Dirk Honecker

Schedule: 12/09/2016 to 16/09/2016

It was proposed to measure the influence of B addition on Co-Re alloys with a nominal composition of Co-17Re-1.2Ta-1.02C at the instrument D33. In addition, a sample with addition of 15 at.% of Cr was also measured. Boron and have a strong influence on the allotropic transformation temperature $\epsilon \leftrightarrow \gamma$ of the Co-Re matrix. Since the Co-matrix phase transformation may come about at the application temperature range of the Co-Re alloys and affects the stability of strengthening TaC precipitates, the exact influence is of interest for the alloy development.

The exact Co-Re matrix transformation temperatures were known from previous neutron diffraction (ND) measurements. In this experiment, the samples were heated higher than the transformation temperature to about 1470 K, hold for 2 h and subsequently cooled to lower temperatures. The evolution of the TaC precipitates was monitored in the in-situ TOF-SANS experiment. The setup allowed a large Q-range with single instrument geometry, which was crucial to monitor the size distribution of the precipitates.

Fig. 1 shows a comparison of the scattering signal at room temperature (RT) in the alloys with composition Co-17Re-0/15Cr-1.2Ta-1.02C after cooling from 1770 K with varying cooling rate and the fitted particle size distributions of TaC precipitates. It can be seen that the scattering signal is generally shifted towards larger Q-values for faster ramps. Therefore, the precipitates have smaller sizes. For comparison, the result of a SAXS measurement with synchrotron radiation and a very fast quenching ramp of 1200 K/min is depicted. Addition of Cr results in much larger precipitate sizes for the smaller cooling ramps.

Fig. 2 shows the scattering signal from kinetic measurements during heating as a color plot. The axes represent temperature vs. Q-vector and the color codes the scattering intensity. The variation of scattering signal originates from a change of the TaC precipitates. In the alloy without Cr, their volume fraction is low in initial state and a strong precipitation is observed upon heating to 1200 K. When heating further to 1690 K, the scattering signal vanishes. This behavior is attributed to a first nucleation at 1200 K and later

dissolution of fine TaC precipitates at 1690 K during the heat ramp. The situation changes in Cr containing alloys where TaC precipitates are already present in initial condition and produce a scattering signal. However, these precipitates dissolve at a similar temperature of 1700 K during heating.

Fig. 3a depicts the SANS curves from kinetic measurements during cooling. These curves were recorded with a time resolution of 15 sec. Although the data is noisy, the nucleation of TaC precipitates can clearly be observed by an intensity increase. In addition, the TaC nucleation can be correlated with the allotropic Co-Re matrix transformation (Fig. 3b), which was measured separately by ND. The scattering invariant (proportional to the volume fraction of TaC) and the radius of gyration were evaluated from the SANS curves and give information about the kinetics and the size of precipitating nuclei.

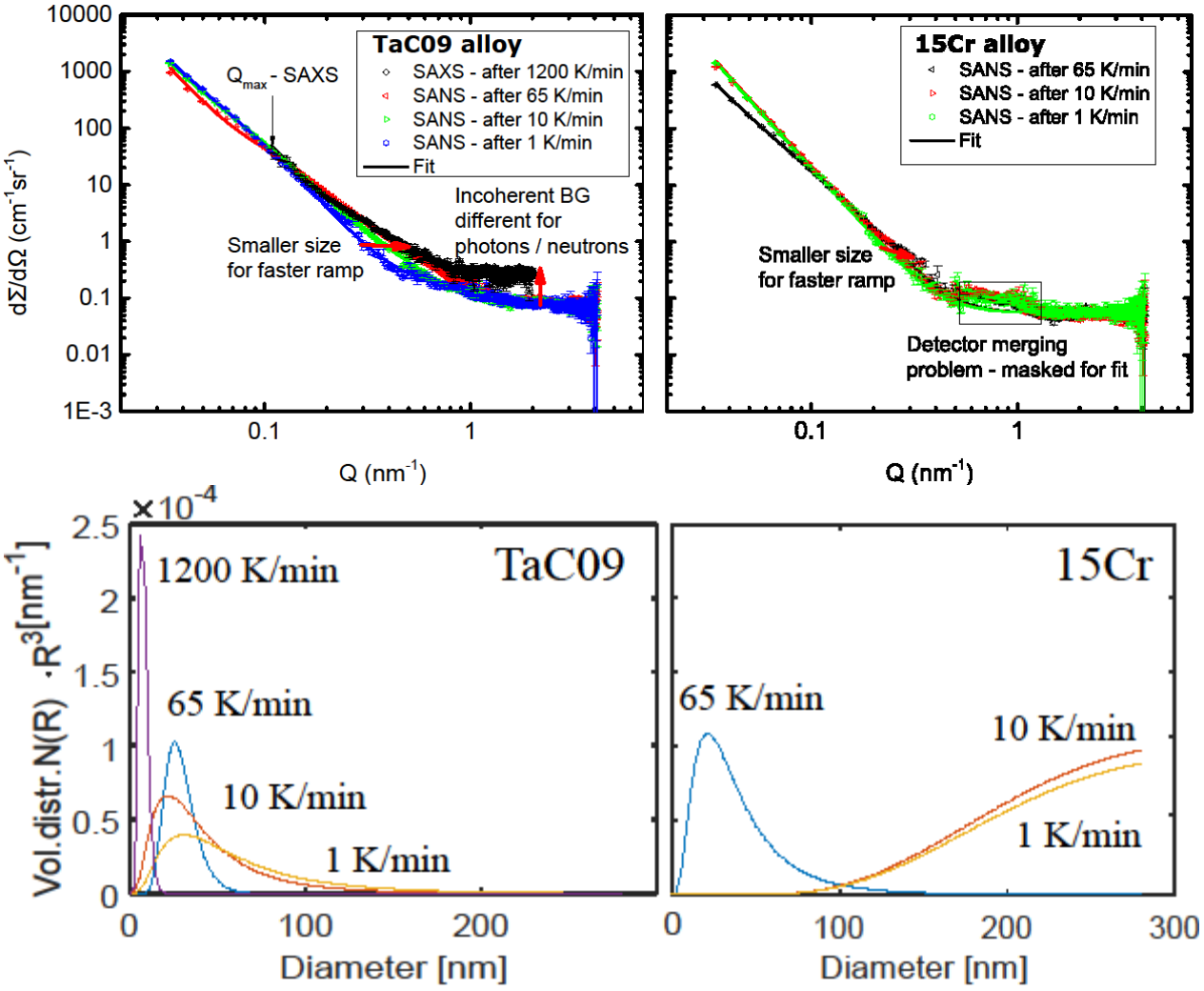

Figure 1: (left) Co-17Re-0Cr-1.2Ta-1.02C, (right) Co-17Re-15Cr-1.2Ta-1.02C. The addition of Cr results in larger particles sizes.

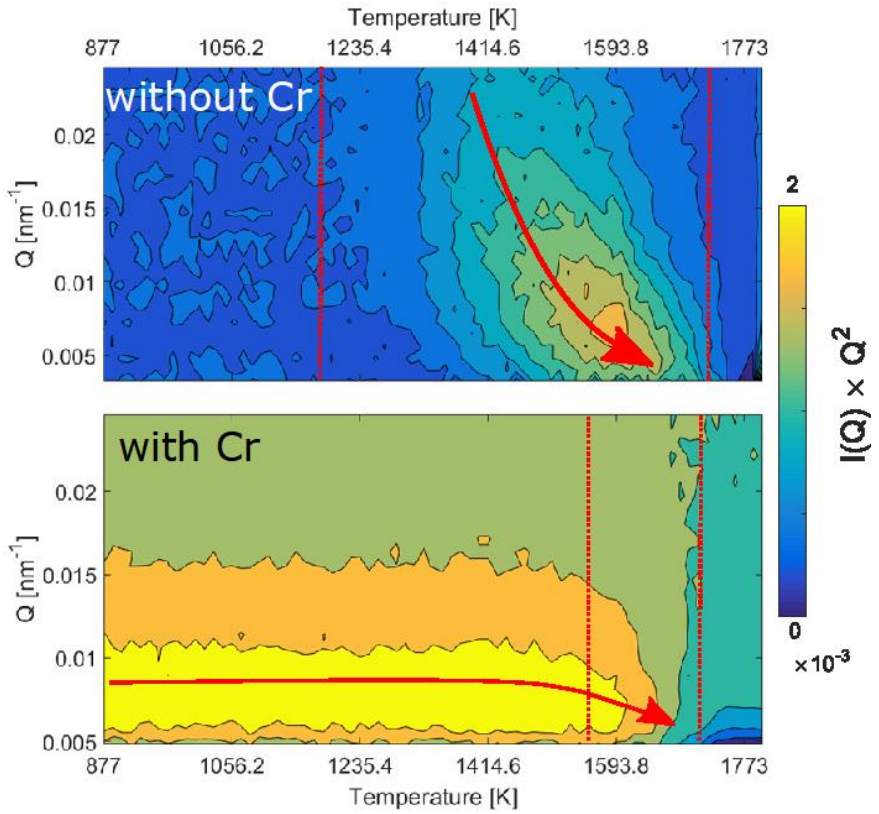

Figure 2: Color plot of scattered intensity vs. temperature and. Q-vector in Co-17Re-0/15Cr-1.2Ta-1.02C alloys during heating. The nucleation and dissolution of TaC precipitates can be observed by a change in the scattering signal.

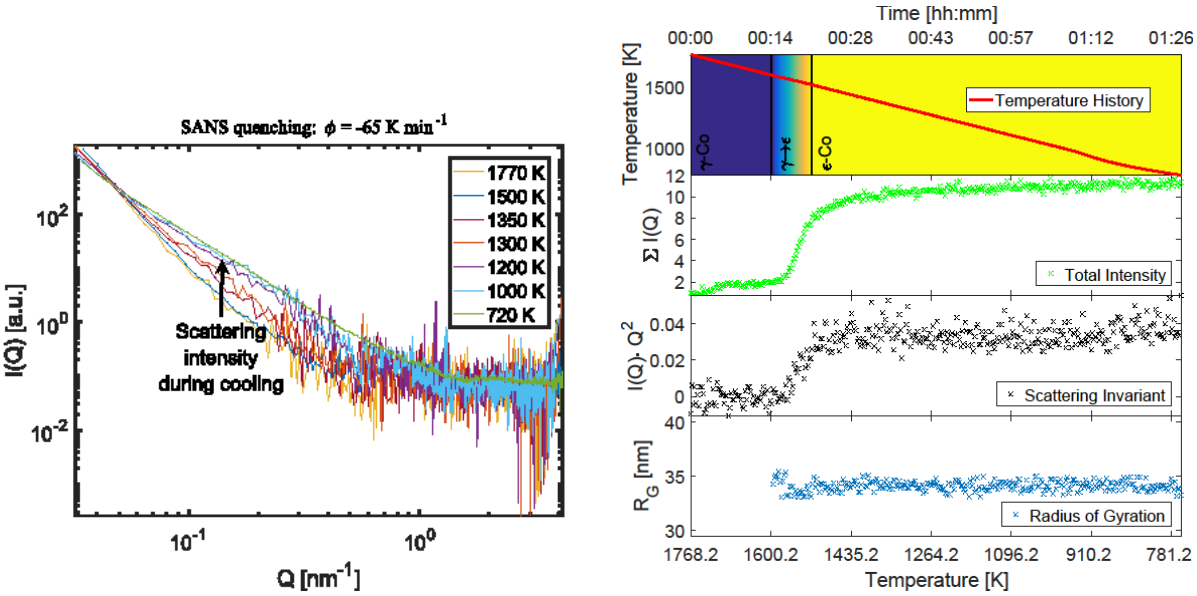

Figure 3: (left) SANS curves during cooling of the alloy Co-17Re-1.2Ta-1.02C. (right) The temperature decrease, scattering intensity, scattering invariant and radius of gyration are depicted. The Co-Re matrix transformation is shown as a color plot and was evaluated from a previously performed ND measurement.