In situ investigation of high temperature corrosion of Co-based alloys in the ESEM - the very first stages

A. Reichmann[2], M. Weiser[3], S. Virtanen[3], P. Poelt[1,2]

[1] Institute of Electron Microscopy and Nanoanalysis, NAWI Graz, Graz University of Technology, Graz, Austria
[2] Graz Centre for Electron Microscopy, Graz, Austria
[3] Institute for Surface Science and Corrosion (LKO), University of Erlangen-Nürnberg, Erlangen, Germany

Introduction

Since the discovery of the ternary γ′-Co_3(Al, W) intermetallic phase with $L1_2$ structure in the system Co-Al-W by Sato et al.[1] cobalt-base superalloys have aroused interest as potential materials for various high-temperature applications.

But one of the still unsolved problems is their resistance to oxidation at high temperatures. To get a deeper understanding of the oxidation mechanisms, the initial steps of oxide layer formation on Co-based alloys were studied *in situ* in the environmental scanning electron microscope (ESEM).

Experimental

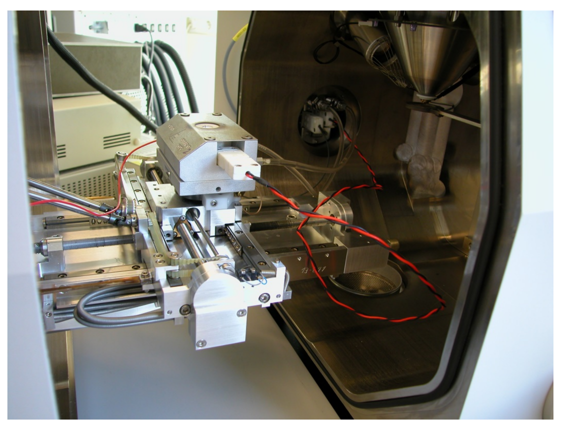
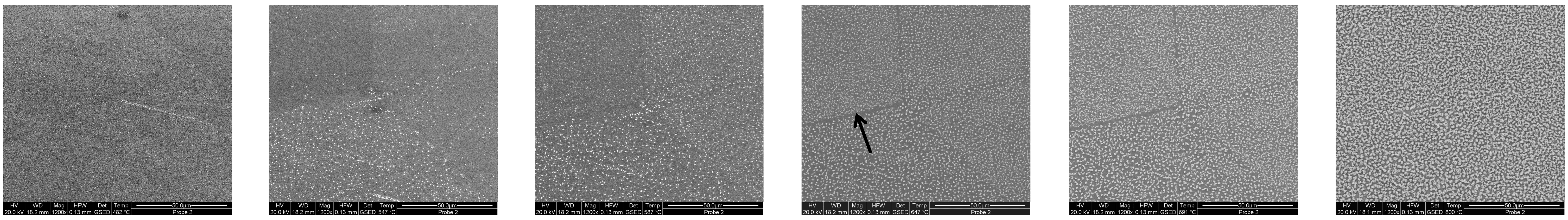


Fig. 1: Image of the heating stage mounted in an ESEM.

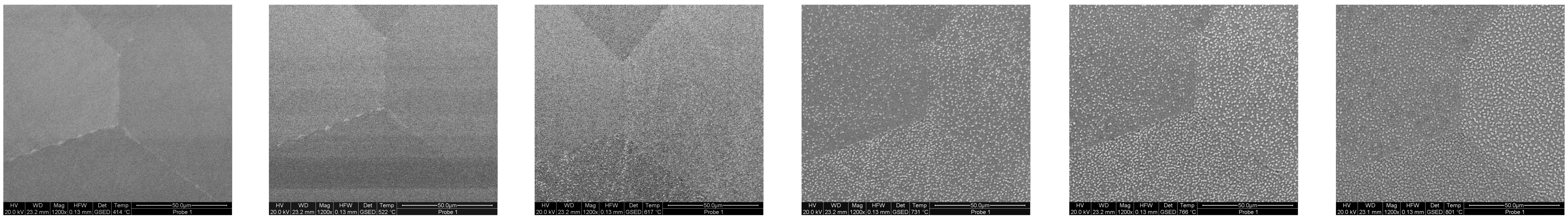
Instruments used for the *in situ* tests:

ESEM: FEI Quanta 600 FEG,
Heating stage: HTS 1000°C (FEI).

Heating tests



482 °C, 65 min | 547 °C, 108 min | 587 °C, 118 min | 647 °C, 148 min | 691 °C, 169 min | 800 °C, 281 min



414 °C, 21 min | 522 °C, 26 min | 617 °C, 32 min | 731 °C, 38 min | 766 °C, 40 min | 801 °C, 48 min

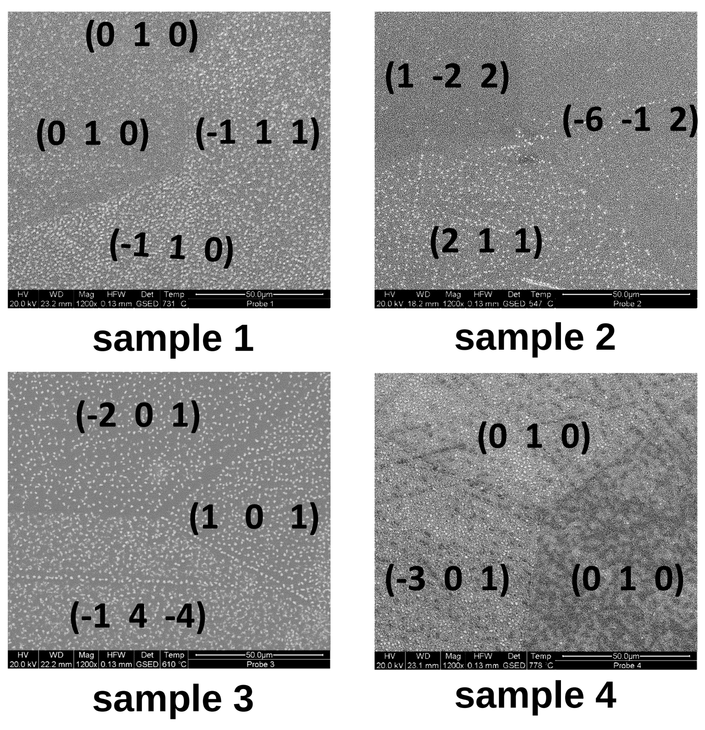
Fig. 2: Co-based alloys (Co-9Al-9W); SE images (image width: 125 µm) of polished samples (colloidal silica) recorded during the heating process at the temperatures given below the images (gas: water vapour; pressure: 133 pascal [~ 5% relative humidity]). Top: temperature ramp of ~ 20 °C/min up to 400 °C, followed by ~ 2°C/min at higher temperatures; bottom: temperature ramp of ~ 20°C up to 800 °C. Subsequently the temperature was held at 800 °C. Time specifications refer to the time elapsed since the start of the heating process. The arrow marks a grain boundary free of corrosion products.

➔ The onset temperature for oxidation, the density of nuclei and the oxidation rate depend all on the crystallographic orientation of the grains.

➔ Oxidation does not always start at the grain boundaries. On the contrary, often grain boundaries free of corrosion products can be found.

➔ The onset temperature of the corrosion process is dependent on the temperature ramp. Thus, variation of the temperature ramp might provide a means to get information about diffusion velocities. Corrosion starts also earlier in case of rough specimen surfaces.

Oxidation and grain orientation



sample	temperature ramp	(visible) start of oxidation	
		T	t
1 (polished)	20 °C/min -> 800 °C	617 °C	35 min
2 (polished)	20 °C/min -> 400 °C 2 °C/min -> 800 °C	482 °C	65 min
3 (polished)	20 °C/min -> 400 °C 2 °C/min -> 800 °C	495 °C	76 min
4 (polished)	20 °C/min -> 800 °C	704 °C	43 min
5 (roughened)	20 °C/min -> 800 °C	492 °C	29 min

Table 1: Approximate onset temperature for oxidation as function of the temperature ramp. The samples 1 - 4 were polished with colloidal silica, thus their surfaces should be nearly stress-free.

Fig. 3: Scale formation in dependence on the crystallographic orientation of the grains.

➔ A clear correlation between the crystallographic orientation of the grains and the development of scales could not be found. The type and number of dislocations still present in the grains might possibly also influence diffusion and thus the start of the oxidation[2]. But grains with a very different crystallographic orientation did not necessarily differ in the oxidation progress.

Scale characterization

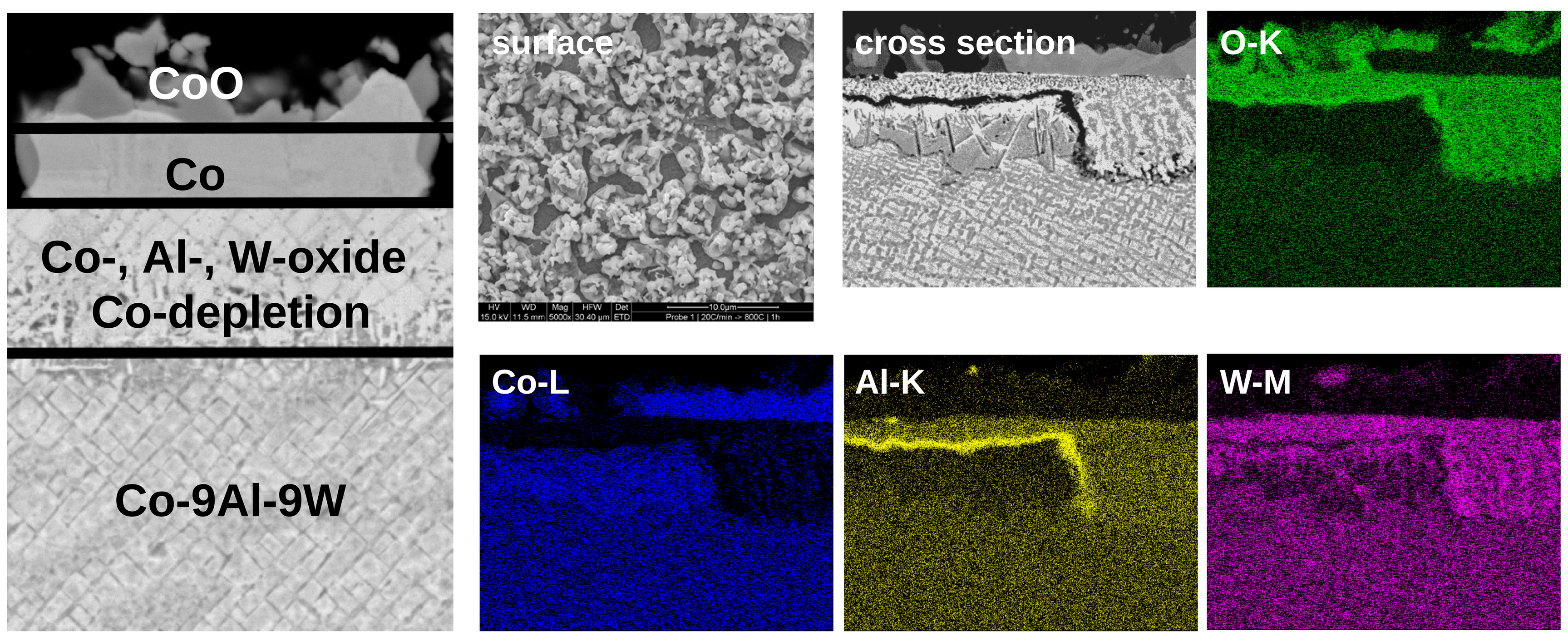


Fig. 4: Co-based alloys (Co-9Al-9W) after heating up to 800 °C (p = 133 Pa, water vapour). Left: BSE image of the cross section of a sample, showing the general structure of the scales (image width: 5.3 µm). Right: SE image of the surface and BSE image of a cross section with the corresponding elemental maps (sample 1, image width: 10 µm).

➔ Under the conditions (especially oxygen activity) used the scales show a layered structure.

➔ At some grains the formation of a dense Al-oxide layer could be observed. It could stop the progress of corrosion.

References

[1] Sato, J., Omori, T., Oikawa, K., Ohnuma, I., Kainuma, R., Ishida, K., Science 312, 90-91 (2006)
[2] Weiser, M., Reichmann, A., Albu, M., Virtanen, S., Poelt P., Adv. Eng. Mat. 17, 1158-1167 (2015)

Contact

Email: peter.poelt@felmi-zfe.at
Homepage: www.felmi-zfe.at