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Influence of the isothermally formed oxide thickness and of the cooling rate on the oxide scale spallation behaviour of chromia-forming Ni-based alloys; thermogravimetry and post-mortem study

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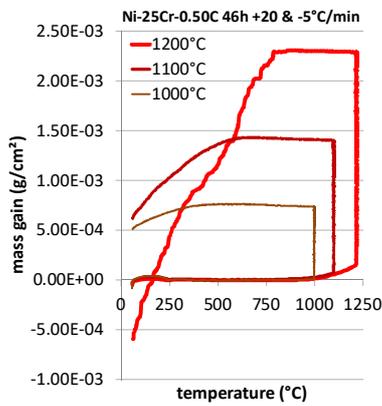
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In addition to the isothermal oxidation rate one must often take into account the resistance of the alloys to oxide spallation when temperature varies to more completely characterize their behaviour in high temperature oxidation [1]. The severity of this phenomenon may be dependent on a lot of parameters as the type of alloy, the minor elements present in its chemical composition, the cooling rate and the different characteristics of the external oxide scales, notably: average thickness, simple or complex constitution, grain size, defects in the scales, presence of water vapour [2,3]... This is thus rather difficult for a given commercial alloy to clearly separate the effects of the too numerous involved factors.

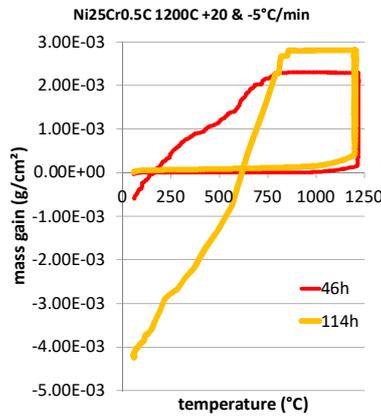
In this work we have focused the study on a ternary alloy, a cast Ni-25Cr-0.5C type which usually behaves as chromia-forming thanks to its high Cr content and the interdendritic chromium carbides network facilitating Cr diffusion, to simplify the constitution of the obtained oxide. Many oxidation tests were undertaken in dry synthetic air with various values of the isothermal stage temperature, of its duration and of the post-isothermal cooling rate. The cooling parts of the obtained mass gain curves were analysed to specify the temperature of spallation start or the temperature decrease before spallation started and the final mass variation (sometimes negative in case of severe spallation), while the percentage of surface coverage of the oxidized samples by the remaining scales was measured by image analysis.

Thermogravimetry curves plotted as mass gain versus temperature

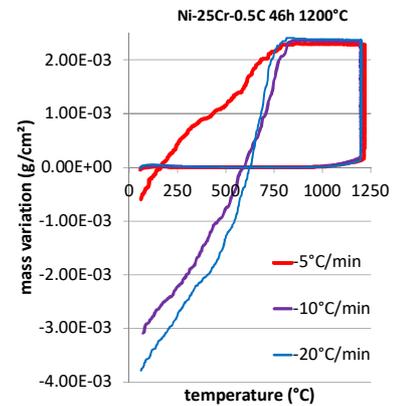
Effect of stage temperature



Effect of the isothermal oxidation duration



Effect of the cooling rate



46h -5°C/min	1000°C	1100°C	1200°C
$T_{spall.start}$ (°C)	423	645	817
mass before spall. (mg/cm ²)	0.75	1.43	2.29
final mass (mg/cm ²)	+0.52	+0.64	-0.59
mass lost by spall. (mg/cm ²)	0.23	0.79	2.88
Fraction of denuded surf. (%)	0.7	5.7	13.8
1 cm			

1200°C -5°C/min	46 hours	114 hours
$T_{spall.start}$ (°C)	817	901
mass before spall. (mg/cm ²)	2.29	2.79
final mass (mg/cm ²)	-0.59	-4.17
mass lost by spall. (mg/cm ²)	2.88	6.96
Fraction of denuded surf. (%)	13.8	15.0
1 cm		

46h 1200°C	-5°C/min	-10°C/min	-20°C/min
$T_{spall.start}$ (°C)	817	885	852
mass before spall. (mg/cm ²)	2.29	2.36	2.40
final mass (mg/cm ²)	-0.59	-3.07	-3.69
mass lost by spall. (mg/cm ²)	2.88	5.43	6.09
Fraction of denuded surf. (%)	13.8	14.4	17.8
1 cm			

Plotting the mass gain, not versus time as usually done for identifying the type of oxidation and for determining the kinetic constants, but versus temperature, allow evidencing the eventual oxide scale spallation at cooling and accessing several parameters characterizing this phenomenon. Here this was the temperature at which spallation began and the amount of lost mass until return to room temperature. The mass gain files recorded during six thermogravimetry tests, carried out for the same alloy oxidized in a same atmosphere, were thus corrected from the air buoyancy variations then plotted versus temperature, in order to specify the effect of several parameters on the behaviour in spallation.

Besides the expected effect of a more or less fast cooling rate (right hand) it appears that the stage temperature (left hand) and the isothermal stage duration (middle) also influences oxide spallation at cooling. The higher the stage temperature or the longer the stage duration is, the higher are the spallation start temperature, the mass loss by spallation and the final surface fraction of denuded surface. Thus these effect are similar to faster cooling rate.

As this is also observed for not more complex Ni-based, Fe-based and Co-based cast alloys [4], the oxide thickness, governed by both the temperature and the duration of the isothermal stage, appears as being a parameter as important as the cooling rate itself.

[1] Young, D., "High Temperature Oxidation and Corrosion of Metals", Elsevier Corrosion Series, Amsterdam (2008).

[2] Berthod, P., Aranda, L., Mathieu ST., Vilasi, M., "Influence of Water Vapour on the Rate of Oxidation of a Ni-25wt.%Cr Alloy at High Temperature", Oxidation of Metals, Vol. 79 (2013), pp. 517-527.

[3] Aranda, L., Schweitzer, T., Mouton, L., Mathieu, SA., Rouer, O., Villegier, P., Berthod, P., Conrath, E., "Kinetic and metallographic study of oxidation at high temperature of cast Ni 25Cr alloy in water vapour rich air", Materials at High Temperature, Vol. 32 (2015), pp. 530-538.

[4] Berthod, P., Conrath, E., "Thermogravimetry and post-mortem study of the oxide scale spallation behavior during post-isothermal oxidation cooling of Cr-rich (Co, Ni or Fe)-based cast alloys with various Hf contents", oral presentation at Eurocorr 2015, Graz.