

EPMA Characterization of Residual Al Content in Oxidized High-Temperature Alloys

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Recent interest in modeling the high-temperature oxidation behavior of alloys in an attempt to better predict their oxidation-limited lifetimes has identified several critical parameters and their influence on the accuracy of the prediction.[1-2] Essentially, lifetime can be correlated to the time required for the concentration at the alloy-oxide interface of the elements required to form the protective oxide scale (usually Cr or Al) to fall to some critical level (C_b) at which protection breaks down. The difference between the starting content and C_b along with the component thickness defines the available reservoir, thus it is essential to determine C_b in order to accurately model oxidation behavior. This critical concentration is different from that calculated from thermodynamic considerations because of the kinetic factors involved in the growth of the oxide scale and interdiffusion in the alloy. Growth of the oxide results in preferential consumption of the element of interest and its depletion from the alloy surface regions. If the rate of removal by oxidation is faster than the rate of diffusion of the element from the bulk of the alloy, then a concentration gradient will develop. However, for alloys designed to form alumina scales at high temperatures, such as Haynes alloy 214 (NiCrAl), FeCrAl-based alloys and iron aluminides (Fe_3Al) [3-4], the rate of diffusion of Al in the alloy typically is faster than the oxidation rate, so that essentially no concentration gradient in Al is developed and all of the Al in the alloy in excess of C_b is available to form protective oxide. The value of C_b potentially changes depending on the alloy composition, microstructure, processing method (cast vs. oxide-dispersed) and exposure temperature.

This study has been undertaken to measure C_b values in various alumina-forming alloys after long-term oxidation exposures which resulted in the breakdown of the alumina scale. Specimens varying in thickness from 0.5-2mm to foils (40-125 μm thickness) were exposed at 1050°-1300°C in air or O_2 in standard cycles either 1h or 100h in duration. After exposure, the specimens were Cu-plated and metallographically sectioned for analysis by electron probe microanalysis (EPMA). Figures 1 and 2 show examples of some of the profiles measured. Table I summarizes the measured C_b values for two commercial oxide dispersion strengthened (ODS) FeCrAl alloys (Special Metals MA956 and Plansee PM200), ingot-processed FeCrAl alloys and an ORNL ODS iron aluminide. In order to improve alumina scale adhesion, the ODS alloys all have an yttrium oxide dispersion and the ingot-processed FeCrAl contains either Y or Ce and La (mischmetal). The ingot-processed FeCrAl had no residual Al content after testing. The full use of the Al reservoir led to exceptionally long lifetimes for this class of materials. The ODS FeCrAl alloys all contained some residual Al content which tended to increase with increasing exposure temperature in MA956. The highest residual Al contents were measured in ODS Fe_3Al which was expected due to its lower Cr content (2at.% versus 20at.% in FeCrAl). However, the surprising result was that the Al profile was not flat in the material (Fig. 2) which needs to be considered in the oxidation lifetime model.

References

- [1] W. J. Quadakkers and M. J. Bennett, *Mater. Sci. Tech.* 10 (1994) 126.
- [2] B. A. Pint, P. F. Tortorelli and I. G. Wright, *Mater. High Temp.* 16, (1999) 1.
- [3] I. G. Wright, B. A. Pint and P. F. Tortorelli, *Oxid. Met.* 55 (2001) 333.

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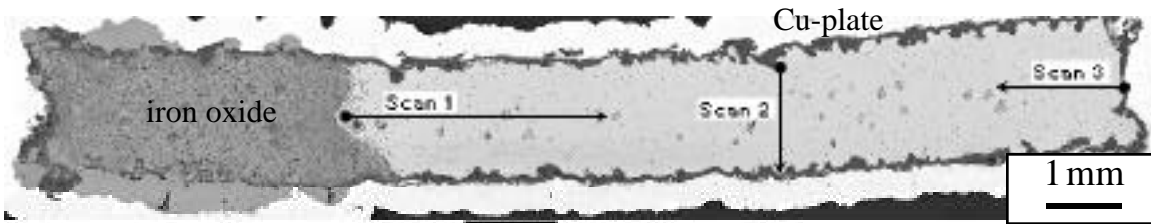


FIG. 1. Light microscopy of polished cross-section of ODS Fe₃Al (Fe-27at%Al-2Cr) oxidized for 28x100h at 1300°C. A thick non-uniform oxide covers the specimen except at the left end where breakaway oxidation has begun oxidizing the entire section. Three EPMA line scans are indicated.

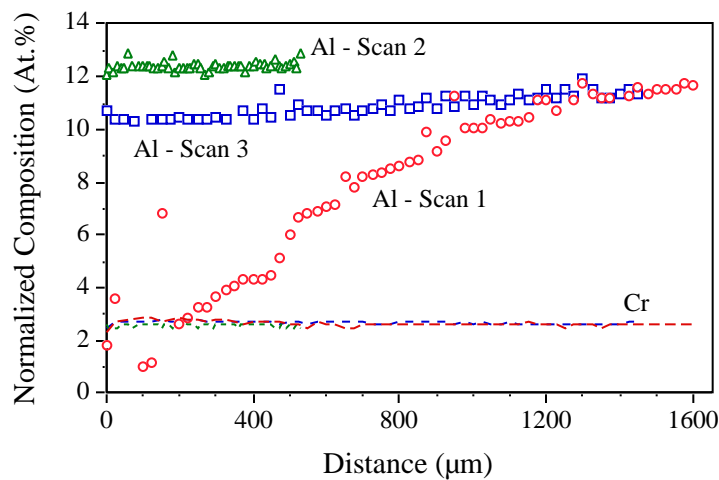


FIG. 2. Residual Al and Cr line scans from the specimen indicated in Fig.1. Near the iron oxide, a different profile was detected. The Cr content was uniform in all three areas.

TABLE 1. Average residual Al content, C_b , for various specimens oxidized to failure at time t_b .

Alloy	Thickness	Temperature	t_b (h)	Cycle Frequency	C_b (at.%)
MA956 ODS FeCrAl	0.5mm	1200°C	350	1h	0.2-0.4
	0.5mm	1200°C	1100	100h	0.3
	0.75mm	1250°C	500	100h	0.3-1.2
	0.75mm	1300°C	300	100h	1.2
PM2000 ODS FeCrAl	40μm	1050°C	350	1h	0.4
	40μm	1100°C	125	1h	0.3
	1.7mm	1200°C	3950	1h	0.5
	1.6mm	1300°C	3100	1h	0.4
FeCrAl + Y or Ce/La	60μm	1050°C	2100	1h	0
	60μm	1100°C	750	1h	0
	1.5mm	1200°C	8800	1h	0
ORNL ODS Fe ₃ Al	1.5mm	1200°C	3950	1h	9.2-10.8
	1.6mm	1300°C	2800	100h	11.5-12.4