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HIGH-TEMPERATURE OXIDATION OF Fe-45%Ta ALLOY – DISTRIBUTION OF SCALES FORMED UNDER DIFFERENT OXYGEN PARTIAL PRESSURES

Rebello, M. C.[1]; Sayeg, I. J.[2]; Garcia F°, J.[3] and Wolynec, S.[1]

[1]Departamento de Engenharia Metalúrgica e de Materiais
Escola Politécnica da Universidade de São Paulo
Av. Prof. Mello Moraes, 2.463 / CEP 05508-900 / São Paulo - SP / Brasil
(011)3818.5241, Fax 3818.5421, *E-mail*: marc@usp.br
[2]Instituto de Geociências da USP / [3]Instituto de Física da USP

The Fe-Ta alloys (15, 30 and 45wt%) were studied in an attempt to develop materials able to resist high-temperature sulfidation[1]. These alloys were obtained from arc melting under argon atmosphere 99.8% tantalum rods (refined in an electron-beam equipment) and 99.98% iron (purchased from Aldrich Chemical). The alloys were heat treated at 1000 °C for 24 hours, followed by another period of 24 hours at 900 °C. The Fe-Ta phase diagram[2] shows that a two-phase microstructure (α-Fe and the intermetallic Fe_2Ta) was expected for all alloys.

The alloy ingots were cut into slices using a diamond saw. After polishing with SiC emery paper (220 – 400 – 600 mesh), samples for oxidation experiments were ready. These samples were oxidized in air and in $CO_2 - H_2$ mixtures (providing oxygen partial pressure of 10^{-24} atm for 600 °C and 10^{-20} atm for 700 °C and 800 °C) in a thermogravimetric analyser (TGA), for periods of 18 (air) and 24 hours (low oxygen partial pressure). The Fe-45%Ta alloy showed important distinctions if one compares the samples oxidized in air with those oxidized under low $p(O_2)$. Kinetic results are summarized in Tables 1 and 2.

After TGA, oxidized samples were submitted to XRD which revealed that the main oxides formed during air oxidation were iron oxides (Fe_2O_3 and Fe_3O_4). For samples oxidized under low oxygen partial pressures no iron oxides were detected but tantalum oxides (mainly Ta_2O_5) and the double oxide $FeTa_2O_6$. Transversal sections of samples were examined at a SEM. The equipment used was a LEICA STEREOSCAN 440, having a tungsten filament, operating with an accelerating voltage of 20 kV, in the BSE mode and a working distance of 25 mm. This examination showed the presence of an internal and an external layer in samples oxidized in air, the latter composed of heamatite, in some cases mixed with magnetite (Figure 1). Under low oxygen partial pressures only internal layers were observed (Figure 2). It was concluded that under low $p(O_2)$ the external scales of Fe_2O_3 and/or Fe_3O_4 are not formed due to thermodynamic reasons, while FeO (which is stable at least for 600 and 700 °C) does not form at all. The EDS microprobe was used to analyse chemical composition of selected areas. This analysis together with stoichometric comparisons confirmed the formation of iron oxides in the external scales after air oxidation (Fe_2O_3 and Fe_3O_4) and showed the presence of double oxides in the internally oxidized zones. For alloys oxidized in air the double oxide analized was $FeTaO_4$, while under low $p(O_2)$ the $FeTa_2O_6$ was detected, in agreement with XRD results.

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Table 1. parabolic constants for air oxidation of Fe-45%Ta

T (°C)	kp ($mg^2.cm^{-4}.h^{-1}$)	r^2
600	0,1419	0,9831
700	0,6435	0,9732
800	Non-parabolic#	0,9968

average slope = 0,847

Table 2. parabolic constants for low $p(O_2)$ oxidation of Fe-45%Ta

T (°C)	kp ($mg^2.cm^{-4}.h^{-1}$)	r^2
600	0,0598	0,9809
700	0,2052	0,9856
800	1,2111	0,9971