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Analysis of AKS- and lanthana-doped molybdenum wire

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Abstract

The lamp industry uses molybdenum wire in many high temperature, structural applications, for which doped molybdenum wire is an important product because it possesses greater high-temperature strength and a higher recrystallization temperature than undoped molybdenum. Prior studies on aluminum–potassium–silicon (AKS) doped tungsten wire have shown that the dispersion which provides the interlocking grain structure in recrystallized tungsten wire is bubbles of elemental potassium; these enhance incandescent lamp filament life. In doped molybdenum the dispersion can be either potassium bubbles, or solid oxide particles, depending on the processing method. Lanthana-doped molybdenum has been reported to have recrystallization temperatures above those obtained through AKS-doping. Lanthana particles are stable within molybdenum to elevated temperatures because lanthanum has very limited solubility in molybdenum. This paper will describe a series of analyses of lanthana-doped and AKS-doped molybdenum wires of a range of sizes.

Keywords: Molybdenum; AKS; Lanthana; Dopant; Recrystallization; Wire

1. Introduction

Molybdenum wire and foil are used extensively in the incandescent lamp industry for coiling mandrels, filament support wires, foil seals, and reflectors for halogen lamps [1–5]. Historically doped molybdenum has been produced in a similar manner to aluminum–potassium–silicon (AKS) doped tungsten, however lower processing temperatures are typically used for the production of molybdenum. The effect of doping molybdenum with potassium, aluminum and silicon is to increase the recrystallization temperature of the molybdenum, and to produce a microstructure of coarse, interlocking grains after recrystallization with rows of fine potassium bubbles pinning the grain boundaries [6].

AKS-doped molybdenum wire is an important product because it possesses improved high-temperature strength and increased recrystallization temperatures; both of these properties are required for incandescent lamp structural applications. In addition, the microstructural stability of AKS-doped molybdenum foil and wire seals during manufacture and operation of quartz envelope lamps, such as quartz-halogen and quartz metal halide lamps, is a major advantage. It is usual practice to employ AKS-doped molybdenum instead of undoped molybdenum in applications where the temperatures are high enough to cause recrystallization and grain growth of undoped molybdenum. In comparison with undoped molybdenum wire, the AKS-doped molybdenum wire possesses a higher recrystallization temperature (~1800 °C for doped wire vs. 1200 °C for undoped wire) and a recrystallized microstructure of coarse, interlocking grains [3–5]. The recrystallized interlocking grain structure provides enhanced high-temperature creep performance [1–3]. The use of AKS-doping of molybdenum has been particularly effective in sheet applications where the sheet furnace elements and hardware can have extended lives [3].

An alternative dopant is lanthana [7–9]. Lanthana-doped molybdenum has been reported to have recrystallization temperatures above those obtained through AKS-doping, as well as a grain structure similar to the coarse, interlocked grains observed in AKS-doped molybdenum [7,8], although little microstructural analysis has been reported on this

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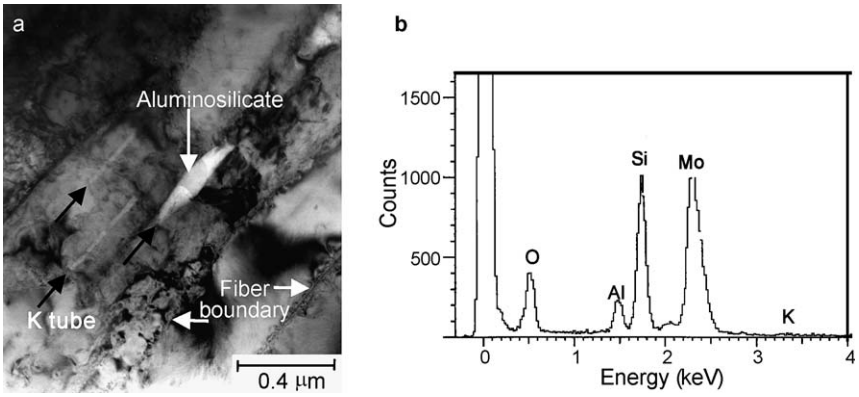

Fig. 1. (a) Transmission electron micrograph of potassium tubes and residual dopants in as-drawn doped 0.41 mm diameter AKS-doped molybdenum wire. (b) EDS spectrum of the aluminosilicate dopant particle shown in (a).

material. Lanthana particles are stable within molybdenum since lanthanum has very limited solubility in molybdenum [10].

2. Experimental

Microstructural and microchemical analyses were performed using analytical transmission electron microscopy (TEM). Analyses were performed on AKS-doped molybdenum wire drawn to diameters of 0.18 mm and 0.41 mm, and on lanthana-doped molybdenum wire from various sources. Three lanthana-doped wires were analyzed, each with slightly different diameters; 0.51 mm, 0.62 mm and 0.64 mm.

The AKS-doped wire was studied in the as-drawn condition, and after recrystallization heat treatments of 2000 °C for 10 min and 2350 °C for 30 min. Lanthana-doped molybdenum wire from three sources was analyzed in the as-drawn condition, after heat treatment at 1800 °C for 30 min, and after heat treatment at 2350 °C for 30 min. Heat treatment of the wire samples was performed in dry hydrogen (dew point <−60 °C) using radiative heating in tungsten tube furnace.

TEM foils of the molybdenum wire samples were made by inserting a short length of the wire into a platinum tube, pressing the tube flat and then mechanically grinding a longitudinal section of the tube until the wire was visible on both sides. The samples were electropolished in a solution of 20% perchloric acid in methanol at −30 °C. Analytical TEM was performed using a JEOL 2010 EM operated at 200 kV with an Oxford ISIS EDS system for X-ray analysis.

3. Results and discussion

3.1. AKS-doped molybdenum wire

Fig. 1(a) shows a bright field transmission electron micrograph of the 0.41 mm diameter AKS-doped molybdenum wire in the as-drawn condition. The wire contained amorphous potassium aluminosilicate particles and some elongated bubbles/ellipsoids that contained elemental potassium. Some of the potassium ellipsoids contained partially decomposed aluminosilicate particles, as can be seen in Fig. 1(a). The compositions of the bubbles and the particles were identified using EDS in the TEM, a typical EDS spectrum is shown in Fig. 1(b). Detection of the potassium tubes, and dopant particles within the fibres and on the fibre boundaries, in the as-drawn wire, was severely limited by the small fibre diameter and the high-dislocation density. However, Fig. 1(a) does show a particle that has been deformed from a sphere into a rod as a result of swaging and wire drawing. The length-diameter ratio is not consistent with the thermomechanical processing area reduction, and this suggests that some break-up/spherodization occurred during thermomechanical processing.

The 0.41 mm diameter AKS-doped wire was not completely recrystallized after heat treatment at 2000 °C for 10 min; there were some residual grains with a size of <20 μm, whereas in the wire that was heat treated at 2350 °C, the recrystallized grains were >20 μm. However, a large degree of recrystallization had occurred and the dislocation density was substantially reduced after heat treatment at 2000 °C. The TEM data showed some remnants of a heavily cold worked structure with well-defined grain boundaries and a high-dislocation density within some grains. Both potassium aluminosilicate and aluminosilicate particles were observed in the wire heat treated at 2000 °C.

Fig. 2 shows a transmission electron micrograph of the microstructure of the 0.41 mm diameter AKS-doped molybdenum wire after heat treatment at 2350 °C for 30 min. The molybdenum matrix was fully recrystallized with grain sizes of >20 μm, and the dislocation density was extremely low, as indicated in Fig. 2. The dopant distribution consists principally of potassium bubbles, as indicated by the EDS analyses. The potassium aluminosilicate particles have all decomposed into potassium bubbles. The aluminosilicate particles have decomposed on heat treatment, since aluminum, silicon and oxygen are soluble in the molybdenum matrix [10].