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Crack initiation mechanism in lanthanum-doped titanium-zirconium-molybdenum alloy during sintering and rolling

Ping Hu [a, b, *], Yu-hang Zhou [a, b], Jie Deng [a, b], Shi-lei Li [a, b], Wen-jing Chen [a, b], Tian Chang [a, b], Bo-liang Hu [a, b], Kuai-she Wang [a, b], Peng-fa Feng [c], Alex A. Volinsky [d]

[a] School of Metallurgy Engineering, Xi'an University of Architecture and Technology, Xi'an 710055, China
[b] National and Local Joint Engineering Research Center for Functional Materials Processing, Xi'an University of Architecture and Technology, Xi'an 710055, China
[c] Jinduicheng Molybdenum Co., Ltd., Xi'an 710077, China
[d] Department of Mechanical Engineering, University of South Florida, Tampa FL 33620, USA

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Lanthanum-doped titanium-zirconium-molybdenum (La-TZM) alloy was prepared by powder metallurgy and rolling process. The processing crack and crack initiation mechanisms of La-TZM alloy after sintering, hot rolling and cold rolling were studied by scanning and transmission electron microscopy. The results show that the doped $La(NO_3)_3$, TiH_2 and ZrH_2 have finer secondary phase in the La-TZM alloy plate. The fracture mode of sintering billet is inter-granular, but the fracture surface of hot rolling exhibited transgranular cleavage. However, the cold rolling is quasi-cleavage fracture. The secondary phase particles tend to hinder the movement of dislocations causing dislocations pile-up. The resulting tensile stress can accelerate cracks nucleation and growth, and the crack propagation will be deflected by the secondary phase particles. The crack initiation can be avoided by reducing local stress concentration caused by dislocations.

1. Introduction

Molybdenum is a refractory rare metal with a melting point of 2620 °C. Molybdenum alloy is widely used in missiles, high temperature heating elements, turbines and fusion reactor components, as well as electrical and electronic manufacturing equipment, aerospace, metal processing, extrusion and forging molds due to its the high strength, creep resistance, thermal conductivity and other properties [1,2]. However, molybdenum has a body-centered cubic crystal structure with less independent slip systems and higher ductile-to-brittle transition temperature, which limit further processing [3–5]. Alloying is an effective way to improve mechanical properties of pure molybdenum and alloys, including TZM and rare earth doped molybdenum alloys. Lanthanum-doped titanium-zirconium-molybdenum (La-TZM) alloy (0.5% Ti, 1% La, 0.06–0.12% Zr and 0.01–0.04% C) has good mechanical properties. Compared with pure molybdenum, La-TZM alloy has high recrystallization temperature, low ductile-to-brittle transition temperature, excellent high temperature strength, low temperature ductility and good welding properties [6–13].

Mechanical properties, high temperature oxidation resistance, corrosion resistance and fracture mechanisms of molybdenum and its alloy have been discussed previously [6–15]. The ultimate strength of the La-TZM alloy is 1405 MPa at room temperature [11]. For the same processing conditions, lanthanum doping has significantly improved tensile strength and elongation, which increased by 28.2% and 32.8%, respectively, and the ductile-to-brittle transition temperature of the La-TZM alloy was decreased to −120 °C, which is 40 °C lower than for the TZM alloy [12]. The recrystallization starting temperature of the La-TZM alloy is 1500 °C, which is 300 °C higher than for the TZM alloy [15]. Only a few research reports focus on the crack initiation in La-TZM alloys during deformation processing. This research group has successfully prepared high-performance La-doped molybdenum by powder metallurgy methods under high temperature and pressure, however, cracks occurred during sintering and rolling. In this paper, the crack initiation mechanism in the La-TZM alloy during sintering and

* Corresponding author. School of Metallurgy Engineering, Xi'an University of Architecture and Technology, Xi'an 710055, China.
E-mail address: huping1985@126.com (P. Hu).

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Table 1
Chemical element composition of pure molybdenum powder (μg/g).

O	N	C	Pb	Bi	Sn	Sb	Cd	Fe	Al	Si	Mg	Ni	Cu	Ca	P	Mo
150	15	5	0.5	0.5	0.5	1	1	5	1.5	2	2	3	1	1.5	1	bal.

Table 2
Sintering procedure of the La-TZM alloy.

Temperature, °C	Heating time, h	Holding time, h
30–400	2	2
400–900	3.5	2
900-1250	2	1
1250–1600	1.5	2
1600–1950	2	3.5
furnace cooling		

rolling was studied by observing microstructure evolution. The purpose of this study is to improve the La-TZM alloy quality during the sintering and rolling process, and to increase production volume.

2. Experimental procedure

The La-TZM alloy plate was prepared by mixing, pressure sintering and rolling. La-TZM powders include pure molybdenum powder with chemical composition listed in Table 1, titanium hydride powder (0.5%), zirconium hydride powder (0.08%), lanthanum trioxide (1%) powder and organic carbon source (fructose). The alloy was doped with Ti and Zr elements using the solid-solid method, and La with C were doped into the Mo-Ti-Zr alloy powder by the solid-liquid method. Then the mixed powders were milled in the planetary ball mill at 40 rpm for 2 h, and the ball-to-powder weight ratio was 2:1, while the diameter of molybdenum balls was 10 mm. Powder was filled into the die mold using the YT79-500 hydraulic press by compression molding with 180 MPa pressure for 20 min. Sintering was performed in the HM3002 intermediate frequency induction furnace in 99.99% hydrogen environment. The temperature of sintering was 1950 °C for 3.5 h. The sintering process had 5 temperature plateaus, and lasted for about 10.5 h. The sintering procedure is shown in Table 2.

The thickness of sintering billet was 12 mm, and the rolling process included hot and cold rolling, and the finished plate thickness was 0.5 mm after cold rolling. The cracks were analyzed in the sintered billet and rolled plate. Several cracked samples of the sintered billet, hot and cold rolled plate after polishing and corrosion testing were observed by scanning electron microscopy (SEM, JSM-6390), and the surrounding components of the crack were obtained from the energy dispersive spectrometry (EDS) data. Microstructure observations of the cold rolled plate were carried out by transmission electron microscopy (TEM, JEM-200CX).

3. Results and discussion

3.1. Cracks microstructure characteristics of the sintered billet

The sintered billet of the La-TZM alloy cracked during the powder metallurgy process. Fig. 1 shows SEM images of the microstructure and the fracture surface of the sintered La-TZM alloy. It shows the whole crack with the source of the crack found in Fig. 1a. Fig. 1b shows that the fracture mode is intergranular

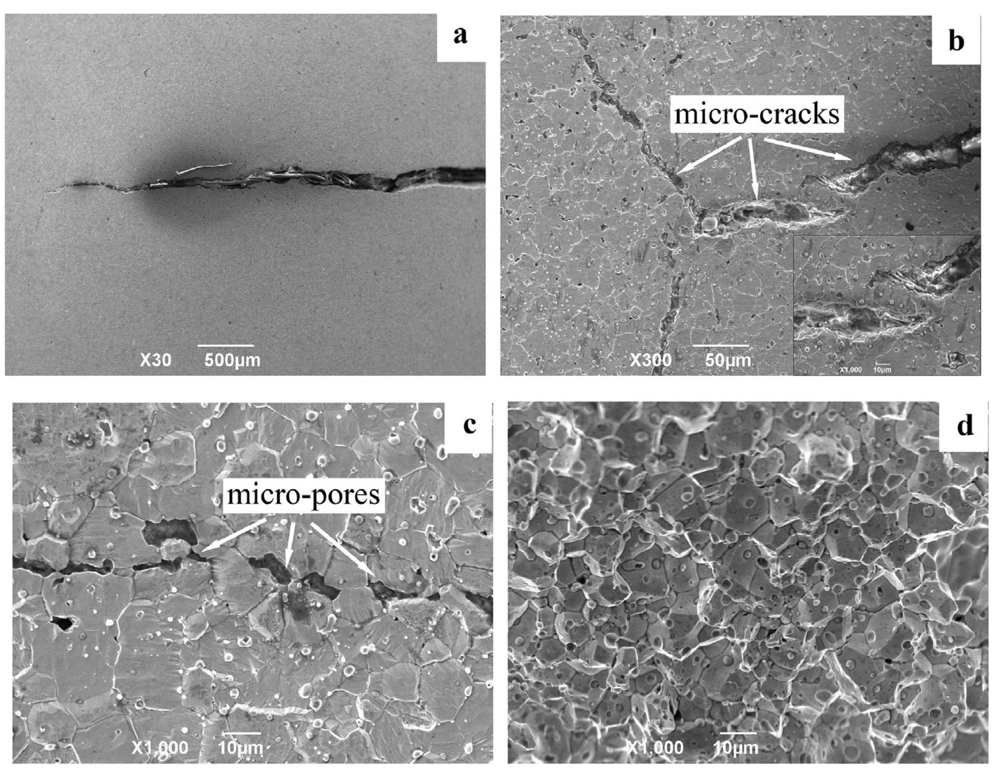

Fig. 1. SEM images of sintering La-TZM alloy crack: (a) macro-crack; (b) micro-crack; (c) micro-pores; (d) fracture surface.