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Enhancement effect of inter-pass annealing during equal channel angular pressing on grain refinement and ductility of 9Cr1Mo steel

Ting Hao [a,*], Haiyin Tang [a], Guangnan Luo [b], Xianping Wang [a], Changsong Liu [a], Qianfeng Fang [a,*]

[a] *Key Laboratory of Materials Physics, Institute of Solid State Physics, Chinese Academy of Sciences, P. O. Box 1129, Hefei 230031, China*
[b] *Institute of Plasma Physics, Chinese Academy of Sciences, P.O. Box 1126, Hefei 230031, China*

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ABSTRACT

To obtain enhanced mechanical property in both the strength and the ductility, 9Cr1Mo steel (T91) was severely deformed by equal channel angular pressing (ECAP) combined with an additional inter-pass annealing. Tensile results show that the additional inter-pass annealing can significantly improve the ductility (i.e. 18% of the total elongation after four-pass extrusion with the inter-pass annealing) but slightly decrease the tensile strength comparing with the case without the inter-pass annealing (i.e. 10% of the total elongation after four-pass ECAP processing). The average grain size of the two passes ECAP-processed materials with the inter-pass annealing ($\sim 0.8\ \mu m$) is smaller than that of the sample without inter-pass annealing ($\sim 2\ \mu m$), and the fraction of the high angle grain boundaries in the samples with the inter-pass annealing ($\sim 40\%$) is higher than that of $\sim 34\%$ (two-pass ECAP) without the inter-pass annealing based on electron backscattering diffraction analysis. The crystallite size and dislocation density were evaluated by means of the *modified* Williamson-Hall plot based on X-ray diffraction analysis. The microstructural analysis indicates that the enhanced ductility of the ECAP processed and inter-pass annealed materials can be attributed to the relatively smaller grain sizes, larger crystallite sizes and lower dislocation densities.

1. Introduction

An enhancement in both the strength and the ductility has been predicted for nanostructured (NS) or ultrafine-grained (UFG) materials over the last several decades based on extrapolations of the grain size dependence of mechanical properties of conventional materials [1,2]. However, the bulk NS and UFG metals generally have relatively high strength but disappointingly low tensile ductility at room temperature (typically less than 2% elongation) [3]. The limited ductility of the NS and UFG metals is main hurdle to the practical utility as the structural materials. Flaws and artifacts from processing, force instability in tension and crack nucleation or propagation were considered as the major limitations to ductility for NC materials [4]. Unfortunately, it is very difficult to avoid the formation of the flaws and artifacts especially when the bulk NS and UFG materials are synthesized by a two-step approach involving the powder synthesis and subsequent consolidation of nanocrystalline powders (e.g., via ball milling or cryomilling) [5].

One-step severe plastic deformation (SPD) processes have been proved to be useful for producing the bulk NS and UFG materials. However, it is also inevitable that a number of the defects such as vacancies or dislocations will be introduced into metals and further evolved to flaws and artifacts like microvoids or microcracks and so on with proceeding accumulation of applied strains during SPD, which lead to the poor ductility of the vast majority of UFG or NC metals. In recent study, some pore- and crack-like defects with micrometer scale were observed clearly in the high-nitrogen austenitic stainless steel subjected to high-pressure torsion processing [6]. As a result, removing or decreasing flaw or artifact size in UFG or NC metals is beneficial to reduce risk of the imposed stress concentration at flaws or artifacts and to suppress the crack nucleation/propagation instability [7]. Several strategies had been proposed to enhance the ductility of NS and UFG metals, such as i) bimodal or multi-modal grain size distribution to gain an increased ductility by resorting the gliding and climbing of dislocations in larger grains; ii) deformation at cryogenic temperatures and/or elevating strain rates to refine the grains and modify the boundary/dislocation structure to increase the uniform elongation [7,8].

On the other hand, appropriate subsequent heat treatment to UFG or NC metals can be expected to obtain both high strength and good ductility. It was reported that an excellent combination of high

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* Corresponding authors.
E-mail addresses: hao.ting@issp.ac.cn (T. Hao), qffang@issp.ac.cn (Q. Fang).

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strength and good ductility can be achieved in the AZ91 Mg alloy subjected to solution heat treatment, ECAP processing and subsequent aging [9]. Al-7Mg alloy with impressive combination of high ductility (elongation ~14.5%) and high strength (UTS ~600 MPa) was obtained by room temperature ECAP combined with inter-pass annealing [10]. It was concluded that the high strength was mainly due to a prominent grain refinement and high dislocation density. While the high ductility was rationalized by an enhanced work hardening originated from the pronounced dynamic strain aging effect and a bimodal grain structure [10]. In this study, we combine equal channel angular pressing (ECAP) method with lateral appropriate low-temperature recovery annealing (inter-pass annealing) to reduce the effect of dislocation interactions (i.e. tangling) on the refinement of the grain size and to decrease flaw or artifact size during each ECAP pass for gaining high mechanical properties. This strategy will result in an advantage to keep a higher strength, on the other hand, it can prolong the uniform elongation (before necking) and increase difficulties for the imposed stress concentration at the reduced flaws to delay the swift fracture failure in tensions in ferritic/martensitic 9Cr1Mo steel (T91). The T91 steel was adopted as the target material because it has been widely used as structural materials for steam generators in nuclear reactors [11].

2. Experimental

The as-received T91 steel (with composition, wt%, 8.63% Cr, 0.23% Ni, 0.95% Mo, 0.43% Mn, 0.003% Ti, 0.21% V, 0.09% Nb, 0.046% Cu, 0.1% C, 0.31% Si, 0.02% P, 0.006% S, 0.03% N) was machined to cylindrical rods of ϕ10 mm × 40 mm in dimension. ECAP processing was carried out in a die with the intersecting angle of 90°. The rods were extruded in a self-designed ECAP die using 63 ton hydraulic press at room temperature. The ECAP setup is illustrated schematically in Fig. 1. All of the ECAP extrusions were conducted in route C, that is to say, after each extrusion the sample was rotated by 180° around the extrusion direction before the next extrusion. For inter-pass annealing processes, the sample after each ECAP processing was additionally annealed at 600 °C for 2 h. This annealing condition has been confirmed only to relax internal strains/dislocation produced by ECAP processing while it does not lead to the recrystallization of the ECAP extrusion samples [12]. The grain size distribution was obtained by analyzing the electron back scattering diffraction (EBSD) patterns recorded using a HKL Nordlys EBSD system interfaced with a field emission gun scanning electron microscope (SEM: Hitachi S-4800). The applied accelerating voltage and working distance were 20 kV and 10 mm, respectively. The EBSD data were analyzed using the software package CHANNEL 5. Grain boundaries (GBs) with misorientation between the contiguous grains of $\theta_{\mathrm{mis}} \leq 15°$ have been defined as low angle grain boundaries (LAGBs), or subgrain boundaries with $\theta_{\mathrm{mis}} < 2°$, and the ones with $\theta_{\mathrm{mis}} \geq 15°$ referred to as high angle grain boundaries (HAGBs). The tensile testing was conducted with Instron 3669 tensile testing machine at a load speed of 0.1 mm/min. The samples with a dimension of 16 mm × 1.5 mm × 1 mm were cut just beneath the surface and the length direction is parallel to the extrusion direction (see Fig. 1). The surfaces of these samples were mechanically polished with standard polishing techniques. The particle size and dislocation density of the samples were studied by X-ray diffraction (XRD) using a Philips Xpert $\theta-2\theta$ powder diffractometer with Cu-Kα radiation (Cu Kα1 radiation, $\lambda=0.15406$ nm). The instrumental broadening was determined and corrected using a standard Si powder.

3. Results and discussion

The engineering tensile stress-strain curves of T91 steel before and after ECAP processing with/without the inter-pass annealing are shown in Fig. 2. The as-received T91 steel exhibits a low ultimate tensile strength (UTS) of ~700 MPa, which is close to ~680 MPa of 9Cr1Mo steel (P91) [13]. The total elongation to failure (TE) of ~31% is also comparable to ~27% of P91 steel [13]. The sample after two-pass ECAP processing (sample A hereafter), UTS increases significantly to ~1030 MPa. However, TE is reduced dramatically to ~17% and the uniform elongation (UE) becomes fairly small (~1%). When the number of ECAP extrusion is further increased to four passes (sample C hereafter), UTS continues to increase up to ~1100 MPa while the TE decreases to ~10%. The behavior of the enhanced UTS accompanying with a reduced ductility due to ECAP processing is consistent with our previous study [14]. For the samples processed by two and four ECAP passes combining with the inter-pass annealing (sample B or D hereafter), the TE (or UE) are significantly increased to ~20% (~7%), and ~15% (~6%), respectively, although their UTS are slightly smaller than that of the corresponding sample A and C.

To understand the mechanical properties of the ECAP-processed samples, their microstructures were characterized by EBSD analysis. Fig. 3(a) displays the EBSD inverse pole figure (IPF) of the as-received T91 steel. The microstructure of the as-received sample consists of prior austenite grains and a number of martensitic laths with a grain size distribution centering at ~21 μm and ~6 μm (inset in Fig. 3(a)), respectively. After two-pass ECAP deformation, the average grain size of sample A decreases significantly to ~2 μm as shown in Fig. 3(b). However, due to the additional inter-pass annealing sample B displays a finer grain size of ~0.8 μm which is smaller than that of sample A as shown in Fig. 3(c). It indicates that the inter-pass annealing has evident influence on the consequent microstructure of UFG materials. Similar result was found in Al alloy that the inter-pass annealing facilitates the formation of nano-sized grains in hard-to-deform Al alloy by room temperature ECAP [15]. Since UTS of sample B with the finer grain size is a little lower than

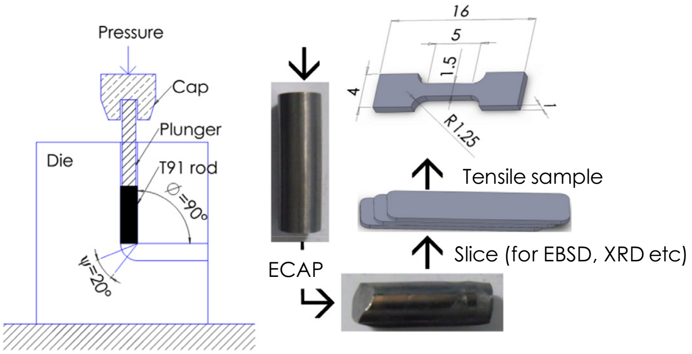

Fig. 1. Schematic of ECAP with a die angle (ϕ=120°) and outer point of contact (ψ=30°). The measurement plane for EBSD and XRD observations and the geometry of cylindrical tensile testing sample are shown as well.