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Effect of temperature on microstructural stabilization and mechanical properties in the dynamic testing of nanocrystalline pure Ti

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ABSTRACT

Commercial purity (CP) Ti was processed by a two-step procedure of ECAP and drawing to give a nanocrystalline (NC) grain size of ~90 nm. Samples were tested in compression at a strain rate of 10 s^{-1} over the temperature range of 298 – 673 K. The results show the high stored energy and high interface energy promote the process of static recrystallization (SRX) when the NC Ti is heated above 573 K. Migration dynamic recrystallization (m-DRX) operates during compressive deformation at temperatures of 298 – 573 K giving a homogeneous microstructure. The compressive yield strength and peak stress at 298 K were measured as 1230 and 1330 MPa, respectively. When the test temperature is increased from 298 to 673 K, the yield strength and peak stress decrease and the work hardening rate and dynamic softening rate are also reduced.

1. Introduction

Over last two decades, ultrafine-grained (UFG) and nanocrystalline (NC) materials have attracted wide attention because of their excellent engineering properties including enhanced strength, good ductility and a superplastic forming capability at elevated temperature [1–7]. Severe plastic deformation (SPD) techniques, such as equal-channel angular pressing (ECAP) [8] and high pressure torsion (HPT) [9], are effective processes for fabricating these materials. In processing by ECAP, the net dimensions of the work-piece are maintained so that the pressing may be repeated repetitively to introduce a very large strain and thereby produce an exceptionally small grain size. In addition, defects such as dislocations may be introduced, and the strength even further improved, by following the ECAP either with more traditional deformation processing methods such as rolling [10], extrusion [11] or drawing [12] or by cutting disks from the pressed billets and processing by HPT [13]. These latter experiments demonstrate that a combination of ECAP and other deformation procedures is often effective in achieving excellent properties in a range of bulk materials having submicrometer and nanocrystalline grain sizes.

The processing of UFG and NC pure titanium is especially attractive because this metal is used extensively both in the aerospace industry and for biomedical applications [14,15]. Much research has been conducted to date on the deformation behavior of UFG/NC Ti processed by ECAP [16–21], HPT [22–24], drawing [25,26] and rolling [27,28] and it was demonstrated that a combination of various post-SPD processing procedures may be utilized successfully for further improving the mechanical properties of pure Ti [29,30]. Nevertheless, there has been no systematic investigation to evaluate the microstructural evolution and the mechanical properties of UFG/NC Ti at high strain rates and elevated temperatures although this information is needed if these materials are used in aerospace applications. Furthermore, although wear resistance is the critical feature generally dominating the use of Ti implants in orthopaedic surgery [31], information is needed also on the dynamic properties in order to successfully design implants to withstand traumatic accidental impacts. In practice, it was shown recently, using machining chips of copper, that the presence of a high fraction of grain boundaries in UFG/NC materials may lead to more complex deformation mechanisms that become especially important at high strain rates and elevated temperatures [32].

Some limited results are now available for the compression testing of pure Ti processed by ECAP and then tested at strain rates up to 10^{-1} s^{-1} and at temperatures from 873 to 1173 K [33].

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However, no results are available documenting the dynamic properties of pure Ti at elevated temperatures after processing by a combination of ECAP and drawing. The present research was conducted to address this deficiency by conducting dynamic tests over a range of temperatures at an imposed rapid strain rate of 10 s^{-1}.

2. Experimental material and procedures

The experiments were conducted on a commercial purity (CP) Ti of grade 2 (ASTM B 348) where the chemical composition is given in Table 1. Billets having square cross-sections of 14 mm^2 were initially processed at a temperature of 473 K using an ECAP-Conform facility. The Conform process was first developed over forty years ago for the continuous extrusion of wire products [34,35] and later an abutment was introduced into the exit channel so that the material was forced through an angle close to 90° to give the ECAP-Conform process [26,36,37]. In the present experiments, all billets were processed at a temperature of 473 K through a total of $N=8$ repetitive passes using an ECAP-Conform facility with an internal angle of 90° so that the imposed strain was ~1 in each separate pass [38]. Immediately following ECAP-Conform, the billets were further processed by drawing to give rods having diameters of 6.3 mm.

Cylindrical compression specimens were cut from the Ti billets after the two-step processing of ECAP+drawing. These specimens had diameters of 5 mm, heights of 5 mm and with their longitudinal axes oriented parallel to the axial or pressing directions of the billets. Dynamic compression was conducted using a Gleeble 3500 thermal simulator at testing temperatures of 298, 473, 573 and 673 K and with an imposed strain rate of 10 s^{-1}. The temperature was recorded in each test using a thermocouple spot-welded to the specimen surface. Prior to compression, each specimen was heated to the test temperature and then held for 3 min to ensure a homogeneous temperature distribution. The flow stress, deformation strain and the strain rate were recorded automatically using this facility. In order to evaluate the overall thermal stability of the microstructure in the ECAP+drawing condition prior to compressive deformation, several specimens of the same size were heated to 473, 573 and 673 K, respectively, held at temperature for 3 min and then the microstructures were observed prior to compression testing.

Table 1
Impurity content in CP-Ti, Grade 2.

Ti	Fe	C	O	H	N
base	0.09	0.01	0.13	0.001	0.01

For microstructural observations, samples for transmission electron microscopy (TEM) were cut from the CP-Ti specimens both before and after the compression testing. These samples were prepared by mechanical grinding using grit papers with different particle sizes from 800 to 2000 mesh and then thinned to electron transparency using a Gatan Dual Ion Milling System. Bright field TEM images were taken using a JEM-2100 LaB_6 microscope operating at 100 kV and selected area electron diffraction (SAED) patterns were recorded from areas of 1 μm^2.

3. Experimental results

3.1. Microstructural evolution

Fig. 1 shows TEM images and SAED patterns in (a) the transverse section and (b) the longitudinal section of a CP-Ti billet processed by ECAP and drawing. In Fig. 1(a) there is a uniform equiaxed microstructure with an average grain size of ~90 nm and examination of the corresponding SAED pattern shows the grain boundaries generally have high angles of misorientation. In Fig. 1(b) there is a banded structure of elongated grains with a high dislocation density and dislocation networks within the grains. Some fragmentation of filamentary grains is also visible in this section and the presence of small arcs in the corresponding SAED pattern demonstrates the presence of internal stresses after processing.

In order to separate the microstructural evolution caused by compressive deformation from the effect of heating to an elevated temperature, the microstructure was observed after heating to temperatures of 473, 573 and 673 K at the same heating rate as used for dynamic compression testing and then holding for 3 min and cooling in air. The microstructures for these three temperatures are shown in Fig. 2 and it is apparent from Fig. 2(a) that there is no significant change when the CP-Ti is annealed at 473 K. Since the NC Ti was processed also at 473 K by the two-step procedure of ECAP and drawing, it can be concluded that the NC Ti is stable up to 473 K. However, as the annealing temperature is increased to 573 K in Fig. 2(b), the elongated grains become less clear and there are some fine new grains, marked by arrows, with sizes of no more than ~50 nm. This shows the development of static recrystallization

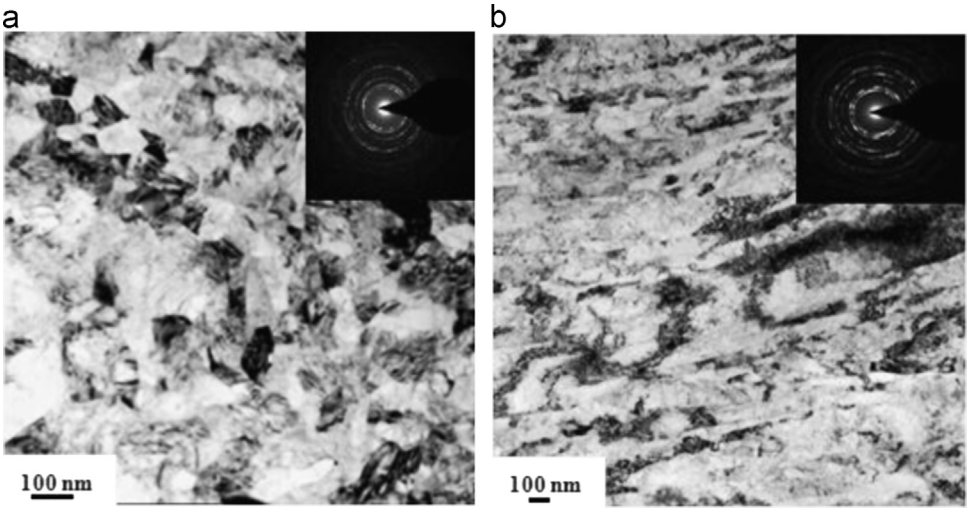

Fig. 1. TEM images and SAED patterns of a CP-Ti billet processed by a two-step procedure of ECAP and drawing (a) in the transverse section and (b) in the longitudinal section.