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Attenuation of ultrasound in severely plastically deformed nickel

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ABSTRACT

Ultrasound attenuation was measured in nickel specimens of about 30 mm diameter prepared using the high pressure torsion technique. The cold working process produced an equivalent shear strain increasing from zero at the center up to 1000% at the edge of the specimen. The fragmentation of the grains due to multiple dislocations led to an ultrafine microstructure with large angle grain boundaries. The mean value of the grain size distribution gradually decreased from ~50 μm at the center to 0.2 μm at the edge. Laser pulses of 5 ns were employed for the excitation of broadband ultrasound pulses covering the spectral range of 0.1–150 MHz. The ultrasound pulses were measured from the opposite side of the specimen by means of an optical interferometer and a piezoelectric foil transducer in two experimental setups. The features of the detected signal forms are discussed. The absolute value of the attenuation decreases from the center to the edge of the specimen showing nearly linear frequency dependence. The variation of the phase velocity was measured in a 6 mm-thick high pressure torsion nickel sample, revealing a velocity increase from the center to the edge.

1. Introduction

The mechanical properties of polycrystalline materials strongly depend on their microstructure. High pressure torsion (HPT) is a relatively new technique employed for severe plastic deformation of metals and composites [1]. It produces huge equivalent strain ε_v, which is proportional to the number of rotations n exerted on a disk-like sample of a thickness d under high hydrostatic pressure. The equivalent strain is calculated as $\varepsilon_v = 2\pi nr/(\sqrt{3}d)$, where r is the distance from the center of the disk [2]. The shear strain increases drastically the dislocation density according to the dislocation multiplication mechanism and induces a grain fragmentation increasing gradually with radius. The reduction in the grain size depends on the material composition and approaches few hundreds of nanometers for metals, after which a saturation of refinement is reached [3]. The refinement of the grain structure increases the yield strength of the materials often without loss of ductility, which is very promising for applications. The nondestructive evaluation of the mechanical properties of such materials is an important task. Different techniques of ultrasound excitation and detection are employed in material sciences for the correlation of the changes of elastic properties and attenuation with microstructure [4–9]. Laser induced ultrasound provides essentially broadband probe pulses from few nanosecond down to tens of picoseconds, allowing a diversity of applications in material science and nondestructive methods [10–14]. The achievable level of elastic strain may involve nonlinear elastic deformation and can be used as the noncontact method for the material strength estimation [15–17]. This study reports on the broadband measurements of the frequency-dependent attenuation of the time-resolved laser induced ultrasound pulses in an HPT nickel sample.

2. HPT nickel sample

The considered HPT-specimen of nickel has a diameter of 33 mm and a thickness of 2.5 mm. The equivalent strain of our specimens linearly increases from zero at the geometrical center up to thousand percent ($\varepsilon_v = 10$) near the circumference. Typical electron back scatter diffraction micrographs taken at zones with defined strain are presented in Fig. 1. The center of the specimen does not demonstrate significant changes of the microstructure and can be considered as polycrystalline material. At the beginning of severe plastic deformation ($\varepsilon_v = 1$, about 1.5 mm from the center for our specimen) the microstructure consists of big grains with large disorientations in their interior. Due to the plastic deformation the dislocation density rises immediately. The dislocations do not arrange randomly. By contrast, they form dislocation cells with very small disorientations. The proceeding deformation increases the disorientations between neighboring cells leading to the occurrence of high angle grain boundaries, more than 15°. At this point new smaller grains appear in the initially coarse structure [1]. The further increase in the shear strain, $\varepsilon_v > 2$,

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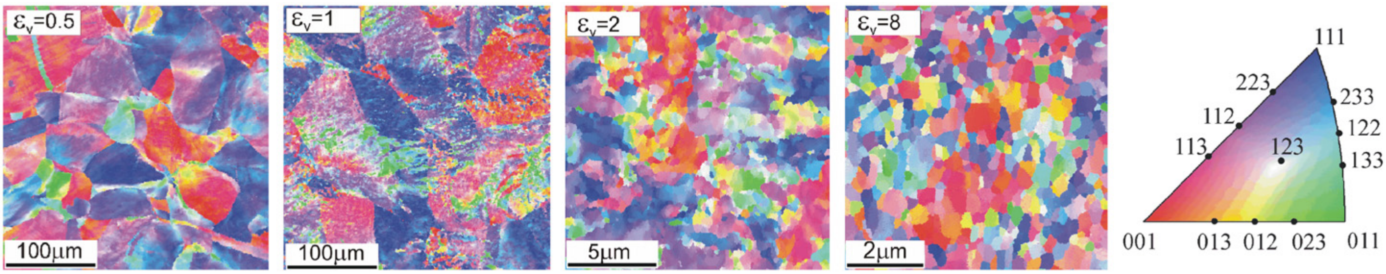

Fig. 1. Electron back scattering diffraction micrographs of the HPT deformed nickel specimen at spots with equivalent strain ε_v. Small angle disorientation within the interior of the grains is observed for the coarse grain structure and a large angle grain boundary is typical for the ultrafine microstructure. The micrographs are obtained for the axial direction, the radial axis is horizontal and the tangential axis is vertical.

induces fragmentation of the small grains that were just developed before. This fragmentation process scales the grain size of HPT deformed nickel down to the hundred nanometers region. The saturation of the grain fragmentation process happens at a strain ε_v of about 8, approximately 12 mm from the center. Grains are elongated in tangential direction with a typical length of 200–300 nm and have a lateral size of 150–200 nm in radial direction. The saturation is given by the balance of the restoration and grain fragmentation processes. Generally, the HPT technique produces an ultrafine microstructure with a typical ultimate grain size of more than 100 nm. This microstructure is intermediate between nanocrystals and polycrystals. A high chemical purity and multiplied dislocation density distinguish the HPT method from others approaching nanocrystalline and ultrafine microstructure.

3. Attenuation of ultrasound pulses

An estimation of the scattering constants was carried out on the base of the unified scattering theory [18] and can be found in Ref. [19] for nickel. This estimation showed that the contribution of dislocation damping to attenuation is the most important effect for the cold working metal and that the phenomenological Granato–Lücke model can be employed [20]. A further development of the theory includes averaging of the dislocation orientation and Burgers vectors but gives similar results [21]. Practically the most important result is that the decrease in the dislocation loop length shifts the maximum of the ultrasonic attenuation to higher frequencies. The variation in the phase velocity depends on the dislocation drag force, which in general is specific for the material and dislocations arrangement. It is noteworthy that there is no comprehensive theory about dislocation dynamics in polycrystals. The said models used limitations of the stress amplitude of elastic waves, as well as low dislocation density, and therefore the models neglect the possible interaction of the dislocations as well as the formation of dislocations as consequence of the interaction.

4. Experimental

The surfaces of HPT nickel samples were mirror-like polished and the planes were parallel within 0.01 mm/cm over the entire specimen. The specimen was mounted onto the side wall window of a water filled bath. A sketch of the experimental setup is presented in Fig. 2. The short transient disturbance is one of the main advantages of the laser ultrasound technique for the evaluation of mechanical properties of materials [13,22]. A pumping pulse of a Nd:YAG laser of 5 ns duration, 1064 nm wavelength and with a pulse energy of < 20 mJ illuminated the water–metal interface. To increase the efficiency of ultrasound generation the surface of the sample was covered by a < 20 μm-thick strongly absorbing layer of

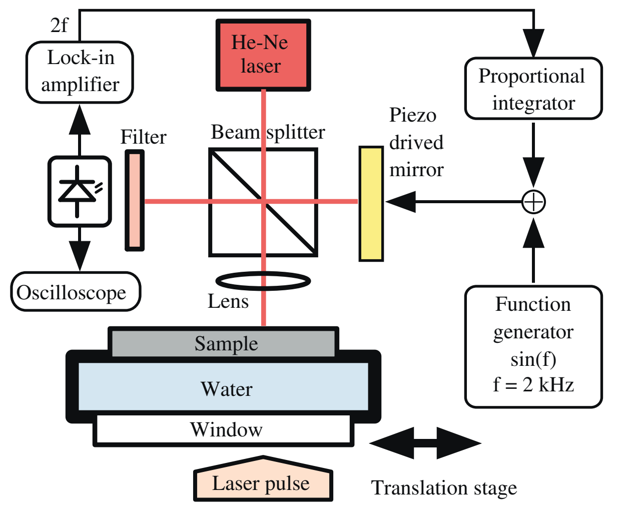

Fig. 2. Sketch of the experimental setup that employs an actively stabilized Michelson interferometer.

black acrylic paint, leading to absorption of most of the laser radiation. The power density on the sample surface was below 15 MW/cm^2 for a diameter of the pumping laser spot of about 6 mm, therefore a maximum pressure pulse magnitude of 15 MPa can be expected [23]. The transient form of the induced pressure pulse is a replica of the envelope of the laser pulse intensity [24]. Thus, a broadband longitudinal pulse with a plane wave front and a pronounced compression phase is launched into the sample.

The optical detection scheme is based on a Michelson interferometer with active stabilization that keeps the operating point at maximum sensitivity for a sufficiently long time for signal averaging [25]. Optical noncontact methods detect frequencies above 100 MHz and possess high lateral resolution on the order of the size of the probe beam spot [10,22]. In the scanning experiment the size of the probe beam spot was estimated as $\delta x = 4\lambda_p F/\,\pi d_b \approx 13\,\mu m$, where λ_p is the wavelength of the probe He–Ne laser, F is the focal length of the focusing lens and d_b is the diameter of the beam. The vertical particle displacement at the surface induces transient changes in the interferometer output beam intensity, which is detected by a photodiode (New Focus) with a bandwidth of 0.1–125 MHz. The power of the probe laser beam of 5 mW and the noise of the detector give a sensitivity of ~0.4 nm to the setup [10].

A series of transient wave signals obtained at different radii from the center of the HPT-sample are presented in Fig. 3. The signals were 256 times averaged at each detection point to reduce the noise of electronics. The primary longitudinal pulse is detected on the surface opposite to the excitation spot at about 0.46 μs after the pumping laser pulse and produces the steeply ascending front of the signal. The ultrasound pulse is completely reflected at the borders due to the large difference of acoustical impedance of air