Parameters Influencing Steady-State Grain Size of Pure Metals Processed by High-Pressure Torsion

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Abstract. High purity elements such as magnesium, aluminum, silicon, titanium, vanadium, iron, nickel, copper, zinc, zirconium, molybdenum, palladium, silver, indium, tin, hafnium, gold and lead were processed by high-pressure torsion and subsequently evaluated by microstructural examinations and Vickers microhardness measurement. The grain size at the steady state, where the grain size and hardness remain unchanged with straining, was determined using either transmission electron microscopy, electron back-scatter diffraction analysis and/or optical microscopy. It is found that the steady state grain sizes are at the submicrometer level in elements with metallic bonding and at the nanometer level in elements with covalent bonding. The correlations between the steady-state grain size and the physical properties of metals are examined and it is found that the atomic bond energy and the homologous temperature are important parameters influencing the steady-state grain size after processing by HPT. A linear correlation between the hardness and grain size at the steady state is achieved by plotting the hardness normalized by the shear modulus against the grain size normalized by the Burgers vector in the logarithmic scale.

Introduction

The significance of grain refinement by high-pressure torsion (HPT) was recognized almost two decades ago by Valiev *et al.* [1]. The principle of the HPT processing is that the sample, in the form of a disc or a ring, is placed between two massive anvils which are rotated with respect to each other under application of compressive pressure, P, to create a torsional strain in the sample [2]. The equivalent strain produced by HPT, ε, is estimated as [3]

$$\varepsilon = \frac{2\pi r N}{\sqrt{3}\, t} \tag{1}$$

where r is the distance from the center of disc (or ring), N is the number of revolutions and t is the thickness of disc (or ring). Earlier reports concerning the HPT processing of pure metals showed that the hardness and grain size in Mg [4,5], Fe [3,6], V [7], Mo [7], Si [8], Al [9-12], Cu [13-16], Ag [17,18], Au [17,18], Ni [18,19], Pt [18], Ti [20,21], Zr [22,23] and Hf [24] reach steady state levels at large strains where the hardness and grain size remains unchanged with further straining.

It was shown that the hardness at the steady state after processing with HPT is determined by shear modulus compensated by homologous temperatures for pure metals [18]. Another paper reported that the hardness at the steady state after processing with HPT is well correlated with the atomic bond energy and related parameters [25]. However, literature survey concludes that little is understood to date regarding the correlations between the grain size at the steady state and the physical parameters and hardness of pure metals after processing with HPT.

In the present study, high-purity elements are processed by HPT and the correlations between the grain size at the steady state and the physical properties are investigated.

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Experimental Procedures

Pure metals and semi-metals of 18 elements (Mg, Al, Si, Ti, V, Fe, Ni, Cu, Zn, Zr, Mo, Pd, Ag, In, Sn, Hf, Au and Pb) with different crystal structures were investigated in this study. The purity levels were 99.9% or higher for the elements. The as-received specimens were cut to discs with 10mm diameters and 0.8mm thickness and HPT was carried out on the discs at room temperature. The disc samples were processed under a selected pressure in the range of P = 1-6 GPa for N = 1/8-10 revolutions with a rotation speed of ω = 0.2-1.0 rpm. The samples after HPT were kept at room temperature for ~30 hours. Thereafter, the samples were polished to a mirror-like surface and the Vickers microhardness was measured from the center to edge at 8 different radial directions and the average values were then plotted against the equivalent strain. The hardness in this study was used from the steady state where the hardness remained unchanged with further straining. The average grain size values at the steady state were determined using either transmission electron microscopy (TEM), electron back-scatter diffraction (EBSD) analysis or optical microscopy (OM).

Results and Discussion

Figure 1 plots the microhardness against equivalent strain for 7 selected metals (Al, Ag, Cu, Pt, Ni, Fe and Hf), demonstrating that all hardness data for each metal follow a unique function of equivalent strain and reaches a steady state at large strains. A micrograph by OM for Sn and an orientation image by EBSD for Zn are shown in Figs. 3(a) and (b), respectively. TEM bright-field micrographs including SAED patterns are in Figs. 3(c), (d), (e), (f) and (g) for Al, Cu, Ni, Fe, Ti and Hf after HPT, respectively. For the 8 selected metals, the grain size varies in a wide range from hundred micrometer level to submicrometer level. The average grain sizes for Sn, Zn, Al, Cu, Ni, Fe, Ti and Hf after HPT are 135, 5.1, 1.9, 0.38, 0.24, 0.20, 0.20 and 0.18 μm, respectively. Since room temperature corresponds to homologous temperatures of 0.59, 0.43, o.32, 0.22, 0.17, 0.16, 0.15, 0.12, inspection of Fig. 2 suggests that the grain size tend to decrease with an increase in the homologous temperature.

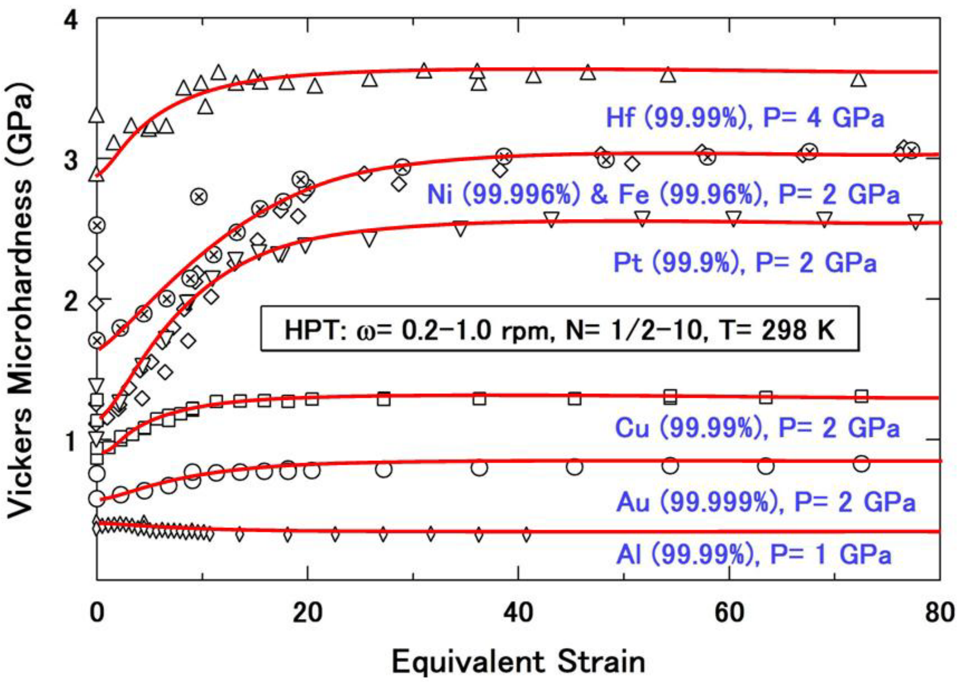

Fig. 1 Hardness plotted against equivalent strain for various metals.

Grain sizes at the steady state (d_S) are plotted in Fig. 3 against the atomic bond energy (ΔH) as attempted in an earlier paper plotting the steady-state hardness against ΔH [25]. The values of d_S are at the submicrometer level (> 100 nm) in elements with metallic bonds and at the nanometer level (< 100 nm) in Si with covalent bonding. Figure 3 shows that d_S decreases significantly with ΔH in metals having low ΔH such as Mg, Al, Zn, In, Sn and Pb, but decreases gradually with ΔH in metals having high ΔH. This trend is different from the variation of steady-state hardness with respect to ΔH where the hardness increases monotonically with an increase in ΔH for all ΔH values [25].

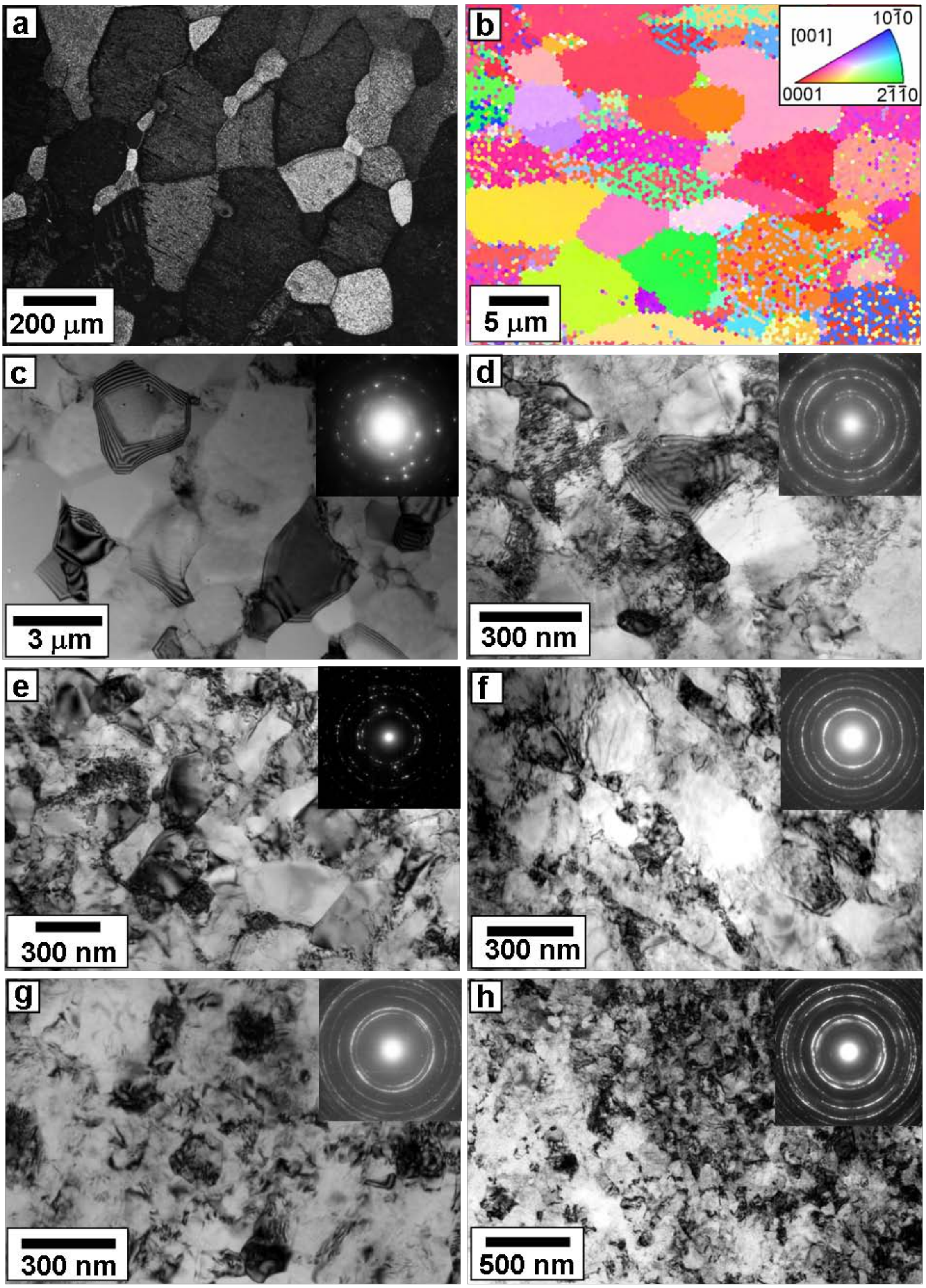

Fig. 6 (a) OM micrograph of Sn, (b) EBSD orientation image of Al; TEM micrographs and SAED patterns of (c) Al, (d) Cu, (e) Ni, (f) Fe, (g) Ti and (h) Hf after processing with HPT at steady state.

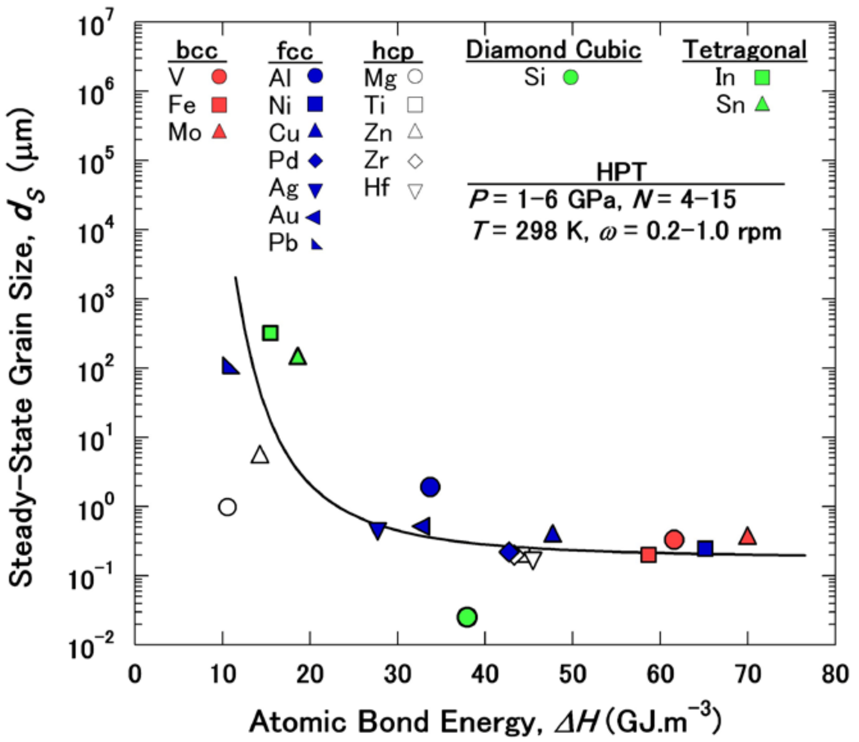

Fig. 3 Grain size at steady state plotted against atomic bond energy.

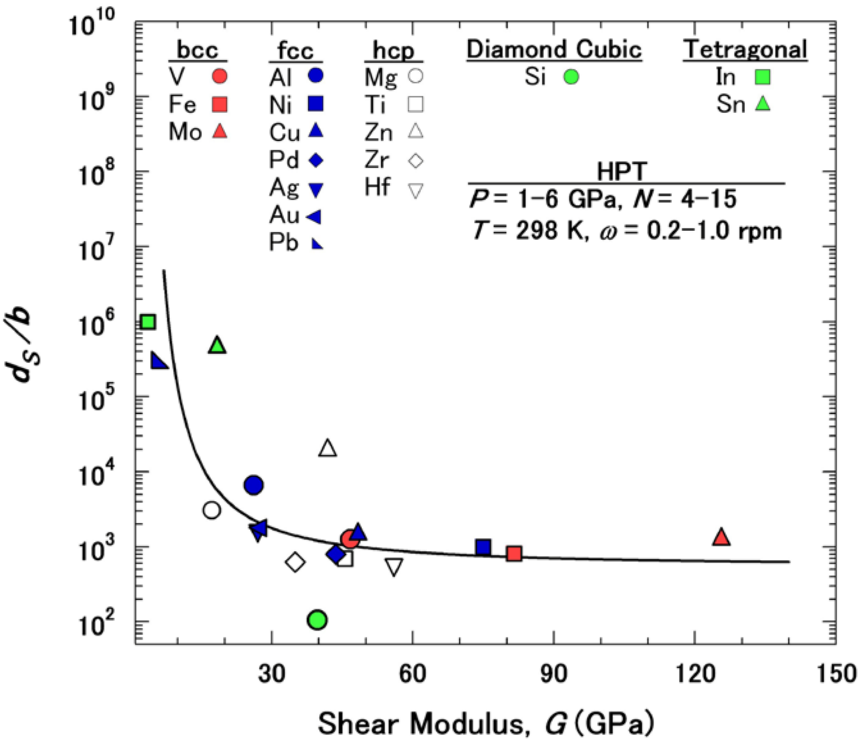

Fig. 4 Grain size at steady state plotted against shear modulus.

Since an earlier study found that the hardness at the steady state is directly proportional to the shear modulus [26], it follows that the grain size at the steady state might be correlated with the shear modulus. Grain sizes at the steady state are normalized by burgers vector and now plotted in Fig. 4 against the shear modulus for elements processed by HPT at room temperature. Figure 4 shows that the grain size tends to decrease with an increase in the shear modulus. However, scattering of the data points appears to be significantly large around the fitted curve, indicating that the shear modulus is not a major controlling factor for the steady-state grain size.

The values of HV_S are normalized by shear modulus (G) and plotted against d_S/b in Figs. 5. It is apparent that all data points lie reasonably on two lines and HV_S/G decreases with increasing d_S/b. Inspection of Fig. 4(a) shows that the following relationship holds between the hardness and grain size

$$\frac{HV_S}{G} = A\left(\frac{d_S}{b}\right)^{-n} \tag{2}$$

where A and n are constants having values of, respectively, 3.38 and 0.63 in metals with high ΔH and 0.7 and 0.21 in metals with low ΔH.

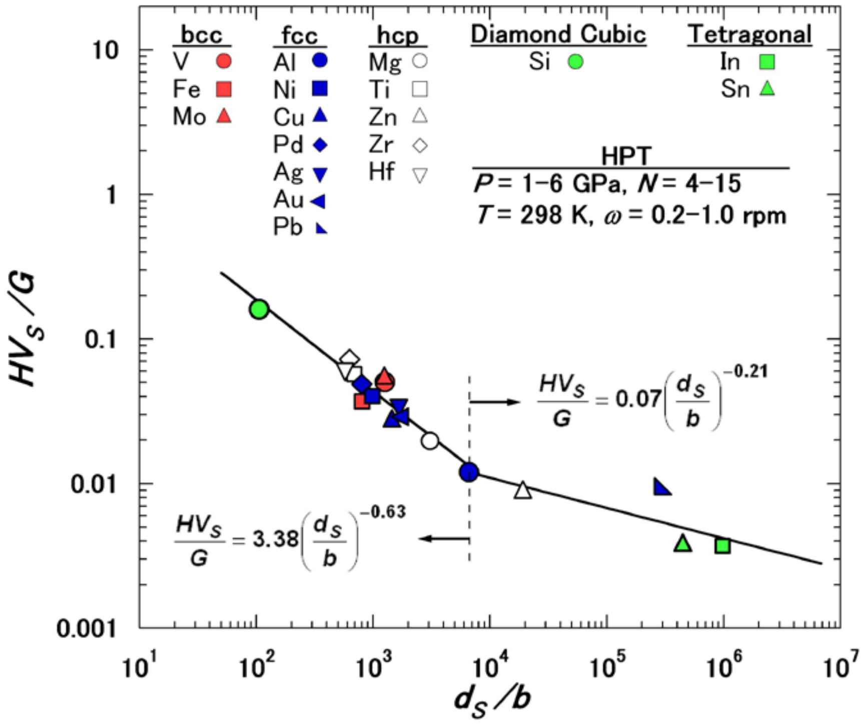

Fig. 5 Plot of HV_S/G against d_S/b. HV_S: steady-state hardness, G: shear modulus, d_S: steady-state grain size, b: Burgers vector.

Conclusions

Metals and semi-metals of 18 elements, with different crystal structures (*bcc*, *fcc*, *hcp*, diamond cubic and tetragonal) were processed by high-pressure torsion and subsequently evaluated by Vickers microhardness measurements and microstructural examinations. The hardness and grain size reach steady-state levels at large strains where the hardness and grain size remains unchanged with further straining. It is found that the atomic bond energy and the homologous temperature are important parameters influencing the steady-state grain size after processing by HPT. The steady state grain sizes are at the submicrometer level in elements with metallic bonding and at the nanometer level in elements with covalent bonding. The grain size decreases with an increase in the atomic bond energy and the homologous temperature. The correlations between the steady-state hardness and the steady-state grain size are also examined and it is found that the hardness normalized by the shear modulus has a good correlation with the grain size normalized by the Burgers vector.

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