сегрегации добавки-стабилизатора.

Математическая обработка зависимостей прочности от давления в рамках соответствующей модели роста трещин может дать важную информацию о прочности и трещиностойкости границ зерен и их соотношении со стандартными значениями при кратковременных испытаниях. Такая информация имеет первостепенное значение при прогнозировании срока службы материалов в тяжелых условиях эксплуатации.

При обеспечении средств контроля длины трещин и развертки во времени при постоянном давлении ГАОВД может быть также использовано для измерения скорости медленного роста при разных нагрузках и составления карт механизмов роста трещины в любых хрупких материалах.

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I. Yu. Prokhorov

FORMATION OF PROPERTIES AND INVESTIGATION OF MECHANICAL STABILITY OF MATERIALS UNDER HYDROABRASIVE COMPRESSION

The behaviour of structural materials, such as steel, corundum and partially stabilized zirconia (PSZ) has been investigated under high-pressure compression through a layer of solid particles. It is shown that steel and Y-PSZ from degranulated coprecipitated powder possess the highest mechanical stability.

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A. Prudnikov[1], A. Misiuk[2], J. Härtwig[3], B. Efros[1], J. Bak-Misiuk[4]

INFLUENCE OF OXYGEN DOPANT IN SILICON ON PRESSURE-INDUCED PHASE TRANSITIONS

[1]Donetsk Phys.& Tech. Institute, NAS of Ukraine
72 R. Luxemburg St., 83114 Donetsk, Ukraine

[2]Institute of Electron Technology
32/46 Lotnikov Al., 02-668 Warszawa, Poland

[3]European Synchrotron Radiation Facility
BP, F-38043 Grenoble, France

[4]Institute of Physics, PAS
32/46 Lotnikov Al., 02-668 Warszawa, Poland

The increase of defect concentration in the hydrostatic pressure (HP) treated Cz–Si samples with initially present SiO_{2-x} precipitates can be considered as a proof of HP-induced massive creation of defects on before-created oxygen-related defects.

An effect of defect creation in the misfitting particle/matrix system has been investigated for about 20 years [1]. Because of importance of silicon as the model semiconductor and basic material for microelectronics, the case of SiO_2 precipitates in Si bulk is of special interest [2].

An attempt to observe the mentioned HP-induced effect of massive creation of «new» defects in Cz–Si with oxygen-related defects was undertaken in this work. An effect of short-time HP-treatment (at HP up to 1.35 GPa) of the oxygen precipitates (OPs)-containing Cz–Si samples at temperatures just below the melting point of silicon (≈ 1680 K) was also investigated.

Experiment

Cz–Si samples were cut from commercially available Cz–Si *p*-type wafers of about 600 μm thickness. The oxygen interstitials concentration (c_0) determined by Fourier Transform Infrared Spectrometry (FTIR) was $\leq 11 \cdot 10^{17}$ cm^{-3}. One sample, designated as *S*, with $c_0 = 11 \cdot 10^{17}$ cm^{-3} was pre-annealed at 1000 K for 20 h to create small oxygen clusters with a total density of $1.3 \cdot 10^6$ cm^{-2} – sample *S*1. In effect of pre-annealing, the c_0 value decreased to $8.5 \cdot 10^{17}$ cm^{-3}. Another sample was pre-annealed at 1000 K for 20 h and, additionally, at 1320 K for 20 h – to create larger defects (OPs with a density of $2.1 \cdot 10^4$ cm^{-2} and stacking faults) – sample *S*2. In effect of such pre-annealing the c_0 value decreased to $6.1 \cdot 10^{17}$ cm^{-3}.

By chemical etching, foils with thickness of 50 μm were prepared from the *S*1 and *S*2 samples. The foils were pressurized for 20–36 hours at 8.5–10.5 GPa at room temperature in diamond anvils cell (DAC), using an H_2O-methanol solution. The DAC design and the pressure measuring device are described in work [3].

Other 600 μm thick, *S*2 and *S*3 samples (for *S*3 initial c_0 was $8 \cdot 10^{17}$ cm^{-3}, *S*3 was pre-annealed at 720 K for 96 h) were HP-treated for 5 min at 1550–1620 K in a high-pressure furnace.

After HP-treatment, the *X*-ray topographs were obtained at the Topography Beamline (ID19) of the ESRF. White beam topography was used for the DAC-treated thin *S*1 and *S*2 samples, and double crystal topography – for the furnace-treated «thick» *S*2 and *S*3 samples. Complementary *X*-ray and FTIR measurements were performed to check structure perfection of the samples.

Results and discussion

The description of defect formation model at the particle/matrix boundary at HP has been presented e.g. in works [2,4]. For a precipitate completely embedded in a matrix, it is necessary to exploit the concept of a misfit (ε) between the matrix and precipitate material. A misfitting precipitate may lose part of its elastic energy when a dislocation is emitted from an interface between the precipitate and the matrix. There are two criteria for estimation of ε and of precipitate size (d) for which dislocation loops can be generated. The first criterion (A) defines the smallest misfit for which the local shear stress at a precipitate boundary can exceed the theoretical shear strength of the matrix. In this case the stress at a precipitate surface and, hence, the critical misfit (ε_{cr}) does not depend on d. The ε_{cr} value for criterion A would be close to $5 \cdot 10^{-2}$ [4].

The second criterion (B) defines the critical misfit from the energy of the system decreases if a dislocation loop is generated at a precipitate boundary, the ε_{cr} value decreases for higher d. Its value would be close to 10^{-3} for $d \approx 1$ μm, and to about 10^{-2} for $d \approx 100$ nm [2,4]. The criterion B applies for generation of dislocations at incoherent precipitates.

«Initial» misfit (ε_0) is present usually at the Si matrix/OP boundary because of much larger OP volume as compared to that of Si as well as of different thermal expansion of Si and SiO_{2-x}.

HP would influence ε at the mentioned boundary because of much higher compressibility γ ($\gamma = 1/K$, where K – the bulk modulus) of SiO_2 as compared to that of Si ($K_{SiO2} = 40$ GPa, $K_{Si} = 98$ GPa). For a spherical embedded particle, the misfit can be estimated from Eq. 1 [4,5]:

$$\varepsilon = \varepsilon_0 + \frac{K_p}{3K_p + 4G_{Si}}\left[\Delta T(\beta_p - \beta_{Si}) + HP\left(\frac{1}{K_{Si}} - \frac{1}{K_p}\right)\right] \qquad (1)$$

where K – bulk modulus; G – shear modulus; β – volume thermal expansion coefficient; $\Delta T = T_{exp} - 300$ K (T_{exp} – temperature for which ε is calculated). The subscripts in Eq. 1 denote the precipitate/matrix.

An estimation (Eq. 1) gives, for SiO_2–Si system at 10 GPa, the misfit value equal to about $1.3 \cdot 10^{-2}$ (close to the value for criterion A, maybe even reaching it under assumption that OPs are composed of stoichiometric SiO_2).

So, at sufficiently high HP one can expect massive creation of dislocations and other defects on each, even coherent, particle. Of course, nonstoichiometry of the OP composition (in comparison to stoichiometric SiO_2) must be taken into account.

White beam topographs of the DAC-treated samples showed elongated, very weak Laue spots. This can be considered as a sign of asterism which has its origin in the HP-induced transformation of Si single crystals to crystals with grains. From this effect it was possible to estimate the mosaic spread of single grains and orientational distribution of the grains. It appears that these mosaic spreads were of the order of 2–6 arc min for individual grains and of 12–30 arc min for the grain distribution (Table 1).

Table 1

Estimation of mosaic spread half-width within individual grain and those of grains within whole crystal

Sample	DAC treatment, GPa	Mosaic spreads, arc min	
		individual grain	grain distribution
*S*1	8.5	2	12
	10.5	4–8	25–30
*S*2	9.0	2–6	20–30

In the case of *S*1 sample (with high density of small oxygen clusters), a strong sample fragmentation after the DAC treatment at 10.5 GPa was observed. The treatment of the *S*1 sample at 8.5 GPa caused much minor structure changes (Table 1).

In the case of *S*2 sample with larger OPs, such fragmentation was observed just after the treatment at 9 GPa. This can be considered as some kind of proof that, at sufficiently high pressure (dependent on *d*), the presence of defects in Cz–Si resulted in massive HP-induced creation of additional defects because of reaching the «criterion *B* value» of ε_{cr}, or, possibly, even the ε_{cr} value according to the criterion *A*.

It must be stressed, however, that it would be impossible to investigate Cz–Si in this respect at higher HP because at about 11 GPa a phase transition occurs in Si [6]. Possibility of some kind of phase transformation at about 10 GPa in amorphous SiO_2 must also be accounted for [7]. In this last case the Si matrix would be untouched, whereas the mentioned phase transformation in SiO_2 (in fact SiO_{2-x} because OPs are typical of substoichiometric composition) would contribute to structural changes at the OP/Si boundary.

In the case of experiments at enhanced temperatures (HT) (at which ε_{cr} is expected to be achieved at lower HP than that at about 300 K), there exists some uncertainty in interpretation of results. It follows, between others, from stress-enhanced oxygen precipitation [8], dissolution of OPs at HT [9] and from structural transformations during sample cooling. So, only short-time HP–HT experiments can lead to conclusive results.

Indications of HP-induced creation of additional defects were found for the samples treated at parameters: ≈ 1580 K – 1 GPa for 5 min. The *A* (10^5 Pa) and *B* (1 GPa) topographs (of the sample *S*2) are quite similar. However, as follows from supplementary measurements, the *S*2 sample structure was more «disturbed» when annealed at higher HP (Table 2).

It can be explained as an effect of «fulfillment» of the conditions for creation of additional defects at the largest defects/matrix boundary (OPs with different sizes were present in the Si matrix).

Table 2

Effect of HP on c_0, on static Debye-Waller factor, L_{660} [9] and on anomalous *X*-ray transmission (*Ia*) for *S* (as-grown) and *S*2 samples treated at 1580 K for 5 min

Sample	HP, Pa	$c_0 \cdot 10^{17}$, cm^{-3}	$L \cdot 10^3$	*Ia*, arb. units
S	10^5	8.6	–	110
S	10^9	8.6	–	120
*S*2	10^5	8.6	26	78
*S*2	10^9	7.9	33	67

In the case of *S*3 samples with small oxygen clusters (created by pre-annealing at 720 K), the effect of HP was even stronger. The treatment at 10^5 Pa resulted in creation of individual large dislocation loops (probably an effect of oxygen precipitation on some structural irregularities during sample cooling), whereas in the samples treated at 1.35 GPa, numerous «additional» defects were observed.

Conclusions

The increase of defect concentration in the HP–HT treated Cz–Si samples with initially present SiO_{2-x} precipitates can be considered as a proof of HP-induced massive creation of defects on before-created oxygen-related defects. However, in the case of DAC-treated *S*1 and *S*2 samples, a misfit dislocation network was not directly proven to be created because of too small sample dimension (all dimensions of about 50 μm) in comparison with the resolution (of the order of a micrometer) of the applied *X*-ray (synchrotron) method. Also, results obtained at HP–HT experiments must be considered with caution, because part of structural transformations can occur during sample cooling (the cooling rate of about 2 $K \cdot s^{-1}$). For this reason, it would be desirable to perform «direct» observations of the HP-induced phenomena by further *in situ* experiments, using DAC mounted directly on the ESRF beam line.

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А.И. Скворцов, В.М. Кондратов, С.Л. Рябов

ФЕРРОМАГНИТНЫЙ РЕЗОНАНС В ДЕФОРМИРОВАННЫХ ДЕМПФИРУЮЩИХ СПЛАВАХ ЖЕЛЕЗА

Вятский государственный технический университет
Россия, 610000, г. Киров, ул. Московская, 36

Обосновано влияние холодной пластической деформации на характеристики ферромагнитного резонанса железа и его сплавов. Проанализирована связь этих характеристик с чистотой деформированного, отожженного материала по примесям, с составом твердого раствора и структурными изменениями при рекристаллизации и полиморфном превращении.