Journal of Solid State Chemistry **154,** 188–190 (2000)
doi:10.1006/jssc.2000.8833, available online at http://www.idealibrary.com on IDEAL®

Physical-Mechanical Characteristics of Fe-Doped Boron

Marina Antadze,[1] Zurab Zoidze, Domenti Gabunia, Otar Tsagareishvili, David Lezhava, and George Darsavelidze

Laboratory of Boron, its Alloys and Compounds, F. N. Tavadze Institute of Metallurgy, Georgian Academy of Sciences, 15, Al. Kazbegi Ave., Tbilisi 380071 Georgia

Recived September 9, 1999; in revised form February 17, 2000; accepted February 28, 2000

The influence of the concentration of iron (0.3 to 3.0 at.%) on the dislocation structure and physical–mechanical characteristics of boron has been studied. It was shown that Fe-doped boron is *p*-type. A hole concentration increase from 6×10^{17} to 2×10^{19} cm^{-3} leads to an increase of the absolute value of the dynamic shear modulus at room temperature from 175 to 190 GPa. The activation energy of the mobility of twin dislocations decreases from 1.30 to 0.75 eV, while those of partial dislocations decrease from 2.1 to 1.8 eV. The interaction energy of twin and partial dislocations with point defects also varies between 0.45 and 0.11 eV. Changes of the structure-sensitive properties observed in Fe-doped boron may be considered as being due to the formation of shallow donor or acceptor levels at defects like kinks on twin or partial dislocations. © 2000 Academic Press

INTRODUCTION

In order to elaborate new technologies for the fabrication of boron-based materials with preassigned physical–mechanical properties, it is essential to study the characteristics of boron doped with various elements. It is well known that Fe atoms may simultaneously occupy different positions in the crystal lattice of boron, forming interatomic bonds of different types with the nearest neighbors (1). Consequently, doping may affect the physical–mechanical properties of boron. For these reasons investigation of the physical–mechanical behavior of Fe-doped boron by the internal friction (IF) method would be of interest from both an analytical and a practical viewpoint.

We have investigated specimens of β-rhombohedral boron doped with Fe (0.3 to 3.0 at.%). The specimens were fabricated using special melting regimes in resistance furnaces. The charges were melted in boron nitride crucibles.

Measurements of the IF temperature dependence were performed in a temperature range from 80 to 1000 K at ~ 1 Hz frequencies on a torsion pendulum. The heating rates varied between 1–3 degree/min. The absolute value of the shear modulus was determined by comparing vibration frequencies of the investigated specimens with the frequencies of a standard one. The Fe concentration was determined with a "Comebax" microanalyzer. The activation energy (H) of the process was measured by the IF maxima frequency shift. The frequency factor τ_0^{-1} was calculated from the condition $\omega\tau = 1$, where $\tau = \tau_0 \exp(H/kT)$ (2). Electrophysical characteristics were estimated from the Hall coefficient and electrical conductivity measurements. The type of conductivity was determined from the sign of the Hall electromotive forces.

Specimens alloyed up to 3 at.% Fe exhibited hole conductivity with the hole concentration variation from 6×10^{17} to 2×10^{19} cm^{-3}. According to Ref. (3), the hole conductivity must change at 2.5 at.% Fe. Such a different behavior may be due to partial electron–hole compensation in the presence of technological impurities.

The IF spectra of Fe-doped specimens were identical and differed only in their activation characteristics. Therefore, only the spectra of two specimens doped with 0.3 and 3.0 at.% Fe are presented (Tables 1 and 2).

In the IF spectrum of boron doped with 0.3 at.% Fe, two low-temperature maxima of relaxation type at 140 and 250 K were observed (Fig. 1, curve 1), characterized by activation energies of 0.15 and 0.45 eV; frequency factors are 1×10^{8} and 5×10^{9} sec^{-1}, respectively. The intensities of the IF maxima increase almost linearly with the increase of vibration amplitude from 5×10^{-5} to 1×10^{-3} accompanied by shifting 10 to 15 K to lower temperatures at higher amplitudes. With the subsequent gradual lowering of the amplitude, the initial characteristics of the maxima are restored.

At temperatures from 80 to 300 K an almost linear decrease of the shear modulus can be observed, except near the IF maximum temperature region where abrupt shear modulus changes were fixed (Fig. 1, curve 1′).

With an increase of the Fe concentration in boron specimens (accompanied by an increase of the hole concentration),

[1] To whom correspondence should be addressed. Fax: (99532)999493. E-mail: root@metall.acnet.ge.

0022-4596/00 $35.00

TABLE 1
Low-Temperature Physical–Mechanical Characteristics of Boron Doped with Fe

Materials	Shear modulus, GPa	Maximum temperature, K	Activation energy, eV	Frequency factor, sec^{-1}	Maximum height, $\times 10^3$	Hole concentration
B + 0.3 at.% Fe	175	140	0.15	8×10^8	0.8	6×10^{17}
		250	0.45	5×10^9	0.5	
B + 1.0 at.% Fe	182	125	0.13	3×10^8	1.3	1×10^{18}
		235	0.38	3×10^9	1.7	
B + 1.8 at.% Fe	185	122	0.12	5×10^7	1.7	3×10^{18}
		230	0.32	1×10^9	2.0	
B + 2.4 at.% Fe	190	120	0.12	1×10^7	2.2	8×10^{18}
		225	0.30	8×10^8	2.4	
B + 3.0 at.% Fe	195	115	0.11	1×10^7	2.7	2×10^{19}
		220	0.28	5×10^8	3.0	

decreases of the IF maximum temperatures and their activation characteristics were found. Simultaneously, the maximum height and shear modulus defect (Fig. 1, curve 2) increased. Physical–mechanical characteristics of boron doped with 0.3 and 3.0 at.% Fe are given in Table 1.

The IF spectrum of boron doped with 0.3 at.% Fe is characterized by intense maxima at 500, 540, 570, and 700 K (Fig. 2, curve 1). The maxima observed at 500, 540, and 700 K have activation energies of 1.0, 1.3, and 2.1 eV and their frequency factors are 8×10^{11}, 5×10^{13}, and 5×10^{14} sec^{-1}, respectively. The maximum at 570 K increases sharply with the increase of the vibration amplitude and has all features of hysteresis type absorption. On the whole the curves of the shear modulus are characterized by a slight linear decrease in the measured temperature range (300 to 1000 K).

Increasing the Fe content in boron specimens causes the following changes in the IF spectrum: the intensities of maxima 140, 250, 500, and 700 K increase, while the maximum temperatures and their activation parameters decrease; defects in the shear modulus at these maxima increase. The intensity of relaxation processes at 540 K sharply decreases without any changes in activation parameters; the IF peak at 570 K is fully suppressed. Activation parameters of the above-mentioned maxima are given in Table 2.

Possible mechanisms explaining the observed relaxation processes may be the following. It is well known that there are a number of polysynthetic twins and stacking faults along the $\{100\}$ planes in the specimens of doped boron (4, 5). This fact, as well as the strong amplitude dependence

TABLE 2
Activation Parameters of High-Temperature IF Peaks of Boron Doped with Fe

Materials	Maximum temperature, K	Maximum height, $\times 10^3$	Activation energy, eV	Frequency factor, sec^{-1}
B + 0.3 at. % Fe	510	2.5	1.0	8×10^{11}
	540	4.5	1.3	2×10^{14}
	710	2.0	2.1	6×10^{14}
B + 1.0 at.% Fe	500	3.2	0.9	5×10^{11}
	530	3.0	1.3	1×10^{14}
	700	2.7	2.0	2×10^{14}
B + 1.8 at.% Fe	485	4.0	0.85	1×10^{11}
	530	2.5	1.3	1×10^{14}
	695	3.2	1.9	8×10^{13}
B + 2.4 at.% Fe	475	3.5	0.8	7×10^{10}
	530	2.0	1.3	1×10^{14}
	690	3.4	1.85	5×10^{13}
B + 3.0 at. % Fe	460	5.5	0.8	2×10^{10}
	530	1.5	1.3	1×10^{14}
	660	3.5	1.8	2×10^{13}

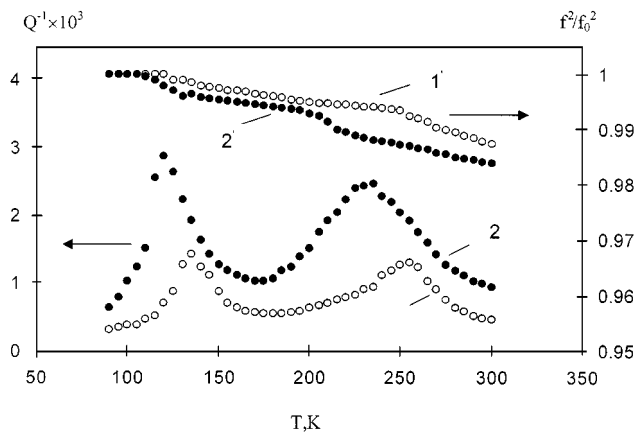

FIG. 1. Internal friction Q^{-1} (1, 2) and relative shear modulus $-f^2/f_0^2$ (1′, 2′) at temperatures ranging from 80 to 300 K in Fe-doped boron. 1, 1′: B + 0.3 at.% Fe. 2, 2′: B + 3.0 at.% Fe.

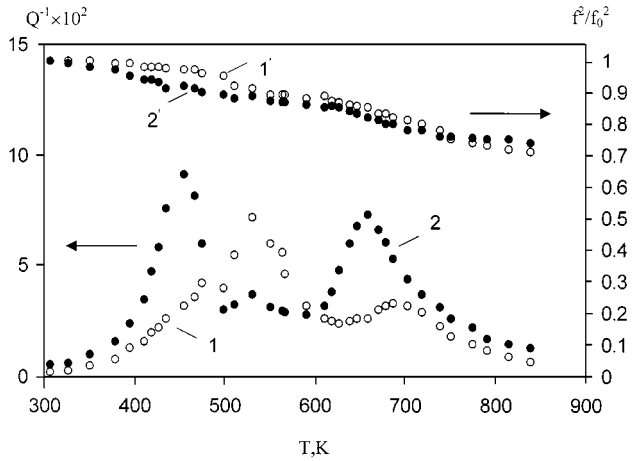

FIG. 2. Internal friction Q^{-1} (1, 2) and relative shear modulus $-f^2/f_0^2$ (1′, 2′) at temperatures ranging from 300 to 1000 K in Fe-doped boron. 1, 1′: B + 0.3 at.% Fe. 2, 2′: B + 3.0 at.% Fe.

of the heights of IF maxima fixed at 140 and 250 K, indicates that they are connected with twin and partial dislocations.

The low-temperature maxima observed in metals and covalent crystals at temperatures from 100 to 300 K are considered to be due to the interaction between dislocations and point defects (6). Relaxation processes in the vicinity of 140 K may be due to the detachment from point defects of single geometrical kinks formed on twinning dislocations.

We observe the maximum at 250 K in quenched undoped and in all doped specimens together with the increase of the $\{100\}$ stacking fault density (4). Therefore, the IF maximum at 250 K may be connected to migration processes of geometrical kinks on partial dislocations in local stress fields of point defects. If we compare the IF spectrum of Fe-doped boron with that of undoped boron (7), then the maxima at 500 and 700 K may be considered as characteristic for doped specimens. These peaks also must be related to twin or partial dislocations.

Possible mechanisms for the explanation of these maxima may be connected with (a) the movement of twinning surface dislocations on (100) planes by creating and broadening dislocation loops (maximum at ~ 500 K) and (b) reversible broadening of stacking faults along the (100) planes under the influence of an external stress field (maximum at ~ 700 K). A hole concentration increase in Fe-doped boron specimens from 6×10^{17} to 2×10^{19} cm^{-3} may cause significant changes in the activation parameters of the IF maxima at 500 and 700 K. Activation energy and prefactor decrease phenomena were found in doped semiconductors by many authors and are explained as the result of the formation of donor or acceptor levels at kinklike dislocation defects (8). The dislocation itself is thought to contain reconstructed bonds only, while there may be dangling bonds at kinks. The same reasoning may be true for partial dislocation kink migration as well as for low-temperature peaks.

Our experiments showed a gradual increase of the shear modulus at 300 K in Fe-doped boron specimens with an increase of the Fe concentration. It was supposed that Fe atoms occupy positions in A(1) holes (9), where they form strong tetrahedral bonds with the nearest surrounding boron atoms. This type of local strengthening of interatomic bonds may cause the observed increase of absolute shear modulus.

REFERENCES

1. J. Callmer, *Solid State Chem.* **23,** 391 (1978).
2. V. S. Postnikov, "Internal Friction in Metals," pp. 352. Metallurgia, Moscow, 1974 (in Russian).
3. U. Kuhlmann, H. Werheit, T. Dose, and T. Lundstrom, "AIP Conference Proceedings," Vol. 231, p. 340. Am. Inst. of Phys., Albuquerque, NM, 1990.
4. G. Sh. Darsavelidze, O. A. Tsagareishvili, T. V. Eterashvili, V. Sh. Metreveli, and D. J. Khomeriki. *J. Less-Common Metals* **117,** 189 (1986).
5. M. J. McKelvy, A. R. Rae Smith, and L. Eyring, *J. Solid State Chem.* **44,** 374 (1982).
6. S. A. Antipov, A. I. Drojjin, I. L. Bataronov, I.V. Mishin, and A. M. Roshchupkin, *Fizika Tverdogo Tela* **31**(9), 189 (1989) (in Russian).
7. G. V. Tsagareishvili, F. N. Tavadze, G. Sh. Darsavelidze, and V. Sh. Metreveli, *J. Electron Technol.* **3,** (1/2, Warszawa, PWN-Pol. Sci. Publ.), 281 (1970).
8. U. Jendrich and P. Haasen, *Phys. Stat. Sol.* (*a*) **108,** 553 (1988).
9. G. P. Tsiskarishvili, T. Lundstrom, G. Lundgrem, G. V. Tsagareishvili, O. A. Tsagareishvili, and F. N. Tavadze, *in* "Proceedings, 9th International Symposium on Boron, Borides, and Related Compounds" (H. Werheit, Ed.), p. 353. Universitat Duisburg Gesamthochschule, Duisburg, 1987.