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Internal friction and anelastic properties of vanadium and V–Ti–Cr alloys

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Abstract

Using an acoustic technique of composite oscillator, the effect of preliminary deformation on the amplitude dependence (amplitude range from 10^{-6} to 3×10^{-4}) of ultrasound damping and the Young's modulus (resonance frequencies) for V and V–(4–10)Ti–(4–10)Cr alloys has been studied. The alloys differed in composition, manufacture technology and also irradiation type (protons, neutrons), environments and fluence were varied. Neutron irradiation (fast reactor, BR-10) of the samples was carried out in argon (irradiation temperature $T_I = 410°C$) and liquid lithium ($T_I = 480°C$) environments. It has been found that, for anelastic deformations of the order of 10^{-8}–10^{-6}, irradiation may result in most cases in noticeable anelastic softening of alloys characterized by a Young's modulus decrease. The softening of the V–4Ti–4Cr alloy irradiated in Ar appeared to be the most significant. The experimental results of acoustic studies might be explained by changing the mobile dislocation point defect interaction, perhaps additionally influenced by existing internal stresses.

1. Introduction

The practical use of V–Ti–Cr alloys as structural materials for fusion reactors requires a comprehensive study of the mechanical properties of the alloys at different levels of micro- and macroplastic deformation. During the studies of acousto-mechanical properties of a V–10Ti–5Cr alloy, two unusual events were found: (1) an effect of Young's modulus decrease (anelastic 'softening' effect) as a result of proton irradiation at room temperature (RT) and (2) an effect of the modulus increase ('hardening') after plastic pre-bending [1].

In this work, the range of V–Ti–Cr alloys was considerably enlarged both in composition and technology of their production; moreover, results of acousto-mechanical tests of neutron irradiated samples are presented here for the first time. Comparison of acoustic parameters of various specimens after pre-straining allowed us to discuss possible mechanisms of the irradiation effect on acoustic properties of the vanadium alloys.

2. Experimental technique

The specimens for acousto-mechanical tests have been manufactured from alloys produced using different technologies in RF and USA. The samples were annealed in vacuum at 1075–1100°C for 1 h. After annealing, the alloys (produced in RF) were irradiated by protons and neutrons. Irradiation by 10 MeV protons was carried out

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at room temperature up to 10^{14}–10^{16} p/cm^2. Irradiation by neutrons (energy > 0.1 MeV) was carried out in the BR-10 fast reactor in argon gas environment (410°C) or in liquid lithium (480°C) up to fluences about 5×10^{21} n/cm^2. Particular data on the samples tested are presented in Table 1; all samples (except neutron irradiated undoped vanadium, sample No. 18 in Table 1) had a form of a bar of approximately 23 mm in length, 1 mm in thickness, and 2–4 mm in width.

The acoustic measurements were carried out by the composite oscillator technique with longitudinal resonant vibrations at frequencies of about 100 kHz. A piezoquartz transducer serving as both a driver and a gauge was included in a bridge circuit. The vibrations of the oscillator, which consisted of the piezoquartz and the tested sample, were at a resonance frequency automatically due to connection of the bridge to the autogenerator circuit. A detailed block-diagram of the acoustic measurement system is given in Ref. [2]. Peculiarities of the set-up in this work consisted, firstly, in the use of the bridge circuit (in this scheme one quartz transducer is used to excite and to detect ultrasonic vibrations) and, secondly, in the use of a PC to collect and to process experimental data, as well as to control the acoustic experiment.

The samples were glued to the piezoquartz, the first harmonic frequency of which was 102 kHz. A longitudinal standing wave was excited in the sample. The strain amplitude range was usually from 10^{-6} up to 3×10^{-4}. The measurements were performed in air at room temperature.

The investigation program included the studies of the amplitude dependence of the attenuation decrement and resonant frequency of acoustic vibrations in samples before and after pre-straining. The measurements started 10 min after deformation. This time was necessary to install the oscillator into the holder; the sample recovered during this period into a new equilibrium state.

Deformation was performed by a three-point bending up to total (elastic and plastic) deflection of 0.6 mm. The distance between the supports of the deformation unit was 19 mm. Application of the load was in the middle of the wide facet of the sample bar. The residual bend values (plastic deformation) were different for the samples; for some of them the plastic deflection was zero. It will be shown below that acoustic experiments with samples of approximately equal dimensions at a fixed value of total pre-straining allow one to demonstrate clearly the effects of different alloy compositions,

Table 1
Influence of pre-straining on internal friction and elastic modulus of vanadium and V–Ti–Cr alloys

No.	Sample	Irradiation	Mass (g)	$\Delta\delta_i$, 10^{-5}	$(\Delta E/E)_i$, 10^{-5}	δ_h, 10^{-5}	$(\Delta E/E)_h$, 10^{-5}
V–4Ti–4Cr							
1	USA	Non-irradiated	0.440	+12.6	+196	+3.9	−7.5
2	V6	Non-irradiated	0.395	+43.0	+152	+19.2	−41.6
3	V6	Neutrons, $T = 410$°C fluence 5.3×10^{21} n/cm^2	0.427	+4.6	−443	+64.9	−214.5
4	V6	Neutrons (Li), $T = 480$°C fluence 4.6×10^{21} n/cm^2	0.416	+67.0	−504	+31.6	−83.5
V–5Ti–5Cr							
5	USA	Non-irradiated	0.430	+20.8	+48.6	+3.1	−11.8
6	V1	Non-irradiated	0.404	+46.9	−156	+16.0	−41.5
7	V1	Neutrons, $T = 410$°C fluence 5.3×10^{21} n/cm^2	0.518	+3.0	−24.0	+1.6	−11.1
8	V1	Neutrons (Li), $T = 480$°C fluence 4.6×10^{21} n/cm^2	0.509	+11.5	−165	+6.6	−15.8
V–10Ti–5Cr							
9	V11	Non-irradiated	0.300	+7.6	−21.8	+0.9	−5.4
10	V2	Non-irradiated	0.404	0	+146	+1.9	−19.2
11	V11	Protons, fluence 2.2×10^{14} p/cm^2	0.300	0	+92.9	+1.7	−8.3
12	V11	Protons, fluence 1.0×10^{16} p/cm^2	0.300	+6.3	+142	+3.3	−7.7
13	V2	Neutrons, $T = 410$°C fluence 5.3×10^{21} n/cm^2	0.254	+15.2	+122	+8.5	−57.0
14	V2	Neutrons (Li), $T = 480$°C fluence 4.6×10^{21} n/cm^2	0.275	+42.8	+80.9	0	−18.2
V (undoped)							
15	V8	Non-irradiated	0.300	+43.2	−32.8	+45.6	−50.2
16	V7	Non-irradiated	0.445	+34.9	−26.0	+50.1	−80.4
17	V8	Protons, fluence 1.0×10^{16} p/cm^2	0.300	+73.0	−465	+49.5	−58.0
18	V7	Neutrons, $T = 410$°C fluence 5.3×10^{21} n/cm^2	1.028	+23.2	−533	+18.6	−46.6

$\Delta\delta_i$, changes of amplitude-independent decrement; $(\Delta E/E)_i$, relative changes of Young's modulus in amplitude-independent damping range at low amplitudes; δ_h, $(\Delta E/E)_h$ amplitude-dependent decrement and Young's modulus defect at vibrational stress amplitude 26.4 MPa.

manufacture technologies as well as irradiation effects on the acoustic properties, of the alloys.

Acoustic parameters measured in this work were: ε is vibrational strain amplitude of a sample; $\sigma = E\varepsilon$, stress amplitude where E is Young's modulus; $\delta(\varepsilon)$, logarithmic decrement of the composite (sample and piezoquartz) oscillator; $f(\varepsilon)$, composite oscillator resonant frequency.

Given the oscillator decrement and resonance frequency, it is possible to calculate decrement δ_o and resonance frequency f_o of the sample using the relations [3]

$$m_o\delta_o = (m_o + m_q)\delta - m_q\delta_q,\ m_o f_o = (m_o + m_q)f - m_q f_q.$$

Here m_o is sample mass, $m_q = 0.52$ g and $f_q = 102\ 000$ Hz, decrement, mass, and resonance frequency of the quartz transducer, $(m_o + m_q)$, composite oscillator mass. Decrement δ_q and piezoquartz resonance frequency f_q do not depend on amplitude. So, the amplitude dependencies $\delta(\varepsilon)$ and $f(\varepsilon)$ of the composite oscillator are defined by the properties of the sample.

Given the amplitude dependence $\delta(\varepsilon)$ and $f(\varepsilon)$, measured before and after pre-deformation, one can determine the parameters listed below, which characterize both elastic and anelastic properties of materials. Among them are

1. Dynamic modulus of elasticity (Young's modulus) E:

   $$E = 4\rho(l_o f_o)^2.$$

   Here ρ is sample material density, l_o, sample length; note that in our tests with vanadium alloys, relative changes of the modulus ($\Delta E/E$), which could appear under the influence of pre-straining and high vibration strain amplitudes, did not exceed 0.6%.
2. Changes in the amplitude-independent decrement $\Delta\delta_i$ and the resonance frequency Δf_i of a sample, as well as amplitude-independent Young's modulus defect $(\Delta E/E)_i = 2\,\Delta f_i/f_o$, which are due to pre-deformation; these parameters are measured at small amplitudes.
3. Amplitude-dependent decrement $\delta_h = \delta_o - \delta_i$, determined as the difference between the decrement δ_o, measured at a given amplitude, and the amplitude-independent decrement δ_i, measured at small amplitudes.
4. Amplitude-dependent Young's modulus defect $(\Delta E/E)_h = 2\,\Delta f_h/f_i$. Here $\Delta f_h = f_o - f_i$ is an amplitude-dependent part of the resonance frequency, measured at a given amplitude.
5. Anelastic vibrational strain

   $$\varepsilon_{an} = (\Delta E/E)\,\varepsilon,$$

   where

   $$(\Delta E/E) = (\Delta E/E)_i + (\Delta E/E)_h.$$

Acoustic experiments, in which the $f(\varepsilon)$ dependence is studied, allow one to analyze both linear anelastic deformation $\varepsilon_i = \varepsilon(\Delta E/E)_i$ and non-linear one $\varepsilon_d = \varepsilon(\Delta E/E)_h$ induced by pre-straining. The dependence $\varepsilon_d(\sigma)$ is due to depinning of dislocations from point defects. It is a real onset of plastic flow which is impossible to observe in usual mechanical (e.g. tensile) tests. The inverse dependence $\sigma(\varepsilon_d)$ yields 'stress–strain' curves, obtained acoustically for a microstrain range.

3. Results

Table 1 presents data on the samples, investigated in this work and their irradiation conditions as well as changes of the amplitude-independent decrement $\Delta\delta_i$ and the Young's modulus $(\Delta E/E)_i$ after pre-straining. The sign 'plus' for the amplitude-independent parameters corresponds to an increase in decrement and elasticity modulus of the sample, and the sign 'minus' to their decrease. As to the amplitude-dependent decrement δ_h and modulus defect $(\Delta E/E)_h$, they arised usually due to preliminary deformation (except some samples); the sign 'plus' for decrement and the sign minus for modulus defect in this case correspond to increasing the damping and decreasing the effective elastic modulus when vibration amplitude becomes higher.

Below, as an example, the results of acoustic tests of the V–4Ti–4Cr alloy in a form of experimental dependence $\delta(\varepsilon)$ and $f(\varepsilon)$ are shown. Further, using the data of Table 1, a description of the experimental behaviour of other materials (including undoped vanadium) is presented as well.

3.1. V–4Ti–4Cr alloy

Results of the tests with the V–4Ti–4Cr alloy sample (USA) are shown in Fig. 1. As one can see from the figure, the decrement and resonant frequency of the sample in annealed state do not depend on amplitude in a rather wide range. After pre-straining, amplitude dependence of the decrement and resonance frequency are clearly seen. A noticeable amplitude hysteresis for the frequency f is revealed: on increasing and decreasing the amplitude, the curves are not coincident. The specific feature of the resonance frequency f behaviour at small amplitudes for this material consists in its increase after pre-deformation.

Fig. 2 presents data for the alloy with same composition, produced in RF. When comparing with Fig. 1, one can see qualitatively almost the same behaviour (f and δ increase after pre-straining and a clear amplitude dependence appears), but it is softer: even in annealed state the amplitude dependence appear both for decrement and frequency; an amplitude hysteresis due to

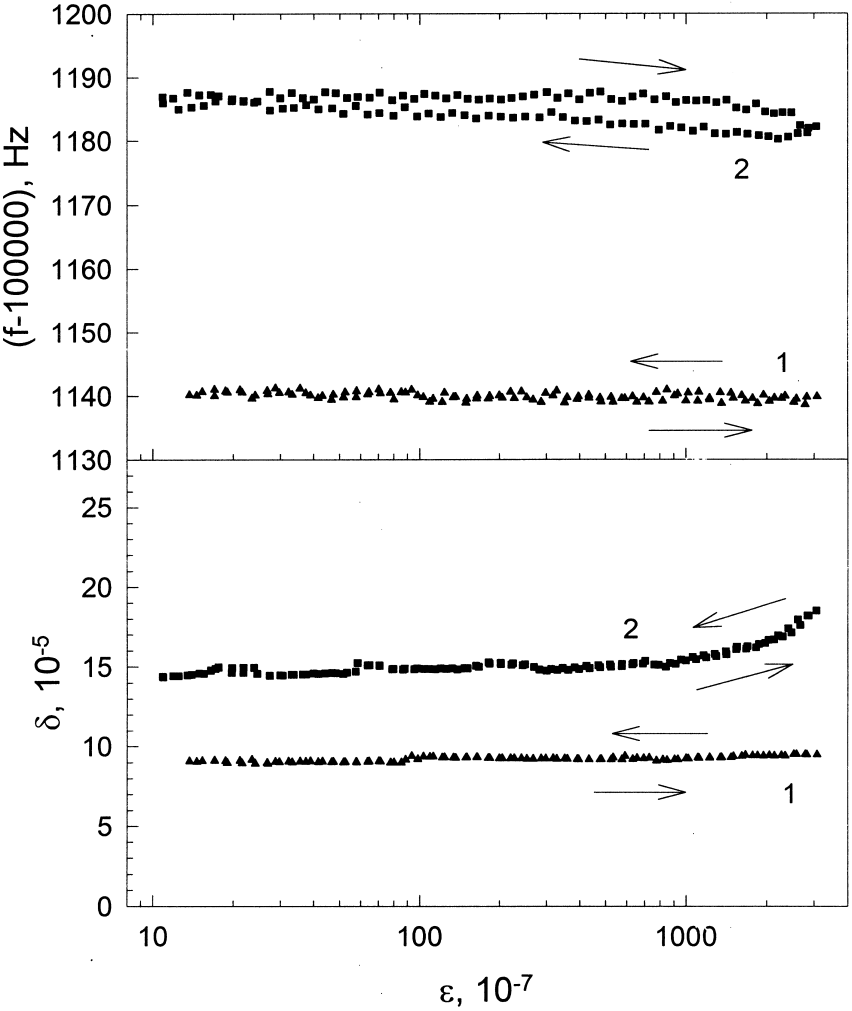

Fig. 1. Amplitude dependence of resonant frequency f and logarithmic decrement δ of non-irradiated V–4Ti–4Cr alloy (produced in USA) before (1) and after (2) pre-straining. The arrows identify the direction of changes in amplitude ε.

deformation is revealed in addition not only for the frequency but for the decrement as well.

Fig. 3 shows the results of tests with the alloy (RF), irradiated by neutrons in argon environment at 410°C. When comparing with non-irradiated material (Fig. 2), it is easy to note some peculiarities: this material, even in the initial state, turned out rather sensitive to an ultrasonic treatment, with the decrement and resonance frequency changing even at the smallest amplitudes; after pre-straining, the resonance frequency (Young's modulus) noticeably decreases against the behaviour of non-irradiated material. However, the amplitude hysteresis hardly changes: mainly a parallel shift to lower frequencies is observed. The decrement changes insignificantly at small amplitudes. As to the amplitude-dependent decrement δ_h at high amplitudes, besides a noticeable growth after deformation, it demonstrates a cross-over effect during increase and subsequent decrease of the amplitude. Note that a cross-over effect was not observed in the same sample for the second and further measurements. Discussion of this effect is given in the next section.

Fig. 4 demonstrates the acoustic characteristics behaviour for the V–4Ti–4Cr sample which was in liquid lithium under neutron irradiation at $T_I = 480$°C. For this sample, the highest values of $\Delta\delta_i$ and $(\Delta E/E)_i$ are observed after pre-straining (Table 1) among all the specimens tested in this work. However, the amplitude dependence behaviour of the decrement and resonance frequency differs considerably as compared with the sample irradiated in argon (see curves 1 in Figs. 3 and 4). This difference may be the result of a fractional anelastic hardening effect, which may be due to nitrogen dissolved in liquid lithium. Nitrogen diffusion into the

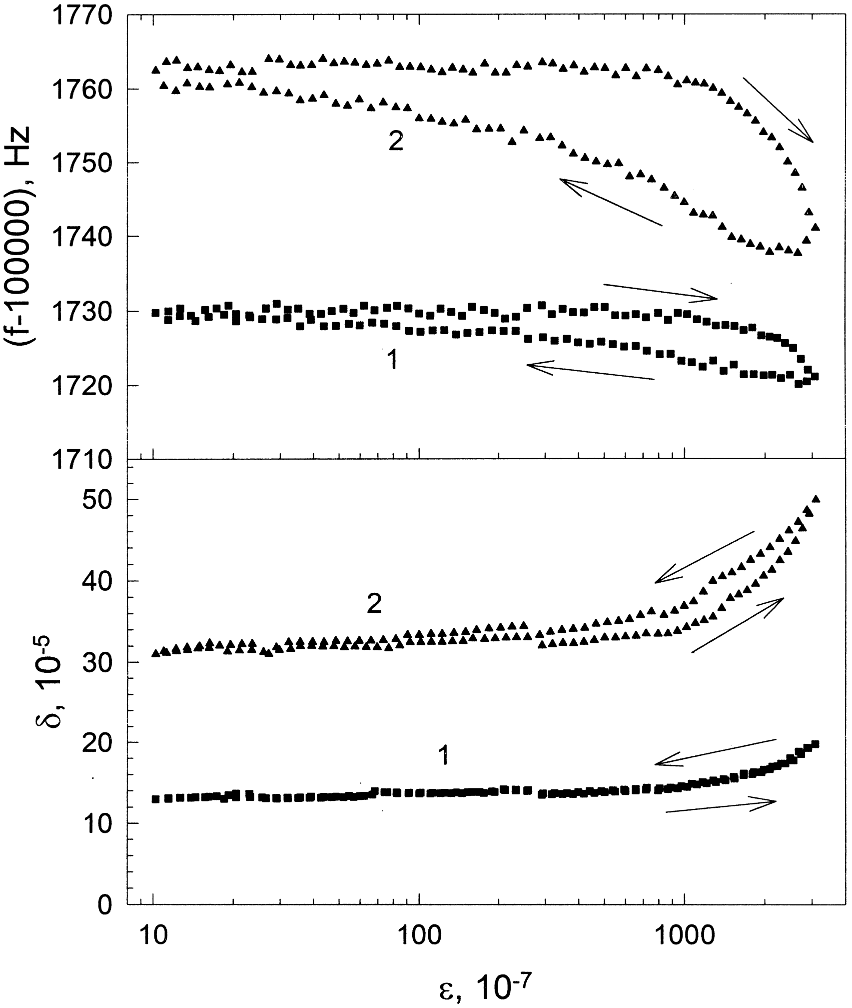

Fig. 2. Amplitude dependence of resonant frequency *f* and logarithmic decrement δ of non-irradiated V–4Ti–4Cr alloy (produced in RF) before (1) and after (2) pre-straining. The arrows identify the direction of changes in amplitude ε.

alloy during irradiation might occur at high temperatures. However, despite hardening probably due to nitrogen impurity, an anelastic softening effect which comes from neutron irradiation is quite noticeable. This is clearly seen from enhanced values of the amplitude-dependent decrement δ_h and modulus defect $(\Delta E/E)_h$ (compare data for samples 2 and 4 in Table 1).

Fig. 5 compares the nonlinear anelastic properties of the V–4Ti–4Cr samples in stress–strain coordinates. The curves of Fig. 5 are obtained from acoustic measurements of vibrational type of loading as against of non-reversible stress–strain diagrams of macroplastic deformation in tensile tests; in the acoustic experiment presented in the paper, an anelastic strain does not exceed the 10^{-6} level, that is 3–5 orders of magnitude lower than that in usual mechanical tests, and this strain is reversible.

3.2. V–5Ti–5Cr alloy

As for the V–4Ti–4Cr alloy, four samples of V–5Ti–5Cr were available: two non-irradiated (1 – USA, 1 – RF) and two neutron irradiated (RF) samples: one was irradiated in argon and one in Li melt.

The behaviour of the acoustic parameters of the American V–4Ti–4Cr and V–5Ti–5Cr specimens are similar, although a quantitative difference exists (Table 1): after pre-deformation the increase of the amplitude-independent decrement is larger and of the Young's modulus, less for V–5Ti–5Cr; the amplitude-dependent parameters are approximately the same.

For non-irradiated materials (RF), the main difference is observed in the resonance frequency behaviour at small amplitudes in the amplitude-independent damping range: while the frequency of the V–4Ti–4Cr sample

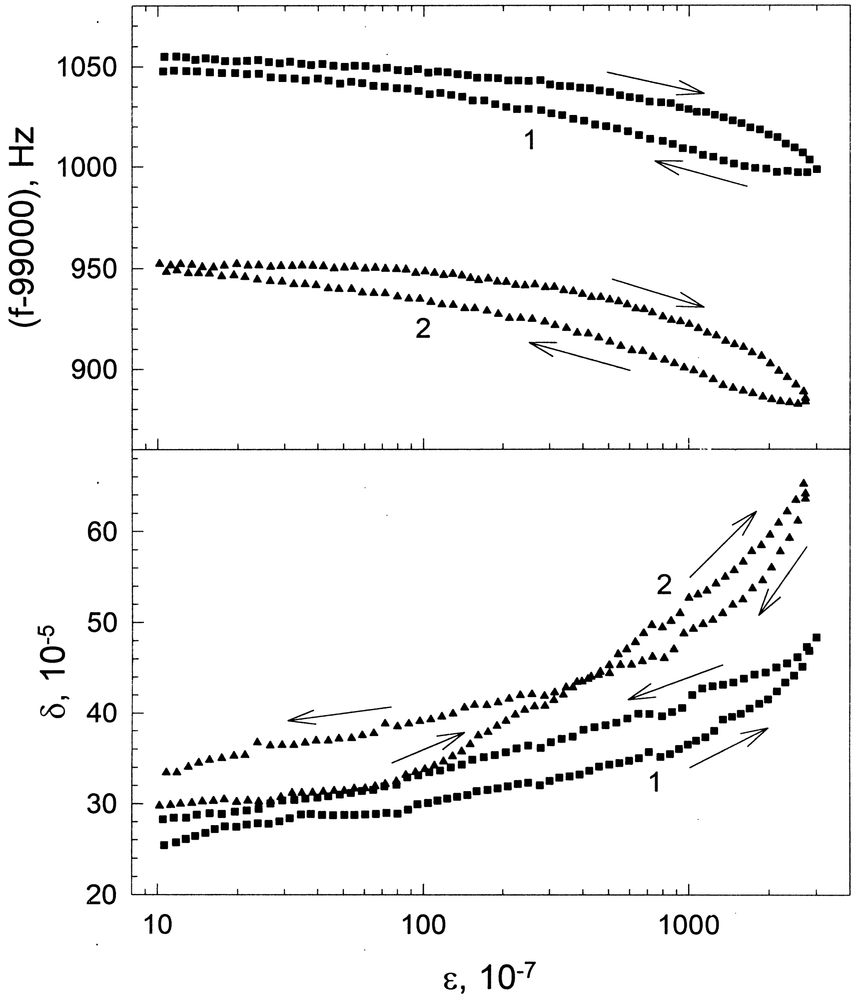

Fig. 3. Amplitude dependence of resonant frequency *f* and logarithmic decrement δ of neutron irradiated V–4Ti–4Cr alloy (produced in RF) before (1) and after (2) pre-straining. The arrows identify the direction of changes in amplitude ε. The sample was under argon during irradiation (410°C).

increases after pre-straining (compare values of $(\Delta E/E)_i$ in Table 1), it decreases for the V–5Ti–5Cr sample. As to the amplitude-dependent decrement and modulus defect, they are almost equal for these samples. A comparison with the samples manufactured by American technology shows that the tendency in changing the acoustic parameters with increase in alloying content conserves here as well. This concerns the changes of the amplitude-independent acoustic parameters after deformation (Table 1). Quantitative difference in the parameters for American and Russian specimens is retained. Note that for non-irradiated samples, the increase in alloy content leads to noticeable softening of Young's modulus and rise in damping after pre-straining in the amplitude-independent damping range and insignificant change in the nonlinear range, where the ultrasonic damping and effective elastic modulus become amplitude-dependent.

Neutron irradiation of V–5Ti–5Cr (Table 1), unlike V–4Ti–4Cr, rather hardens the material, although some increase in the negative value of $(\Delta E/E)_i$ is observed for the sample irradiated in liquid lithium. Note that the neutron irradiated V–5Ti–5Cr samples had almost no residual deflection. This anelastic hardening due to neutron irradiation of the V–5Ti–5Cr alloy for nonlinear parameters is clearly demonstrated in Table 1 by the amplitude-dependent decrement and modulus defect data. It is interesting to note that for this material, the sample irradiated in liquid lithium is softer than the sample irradiated in argon, unlike the V–4Ti–4Cr alloy.

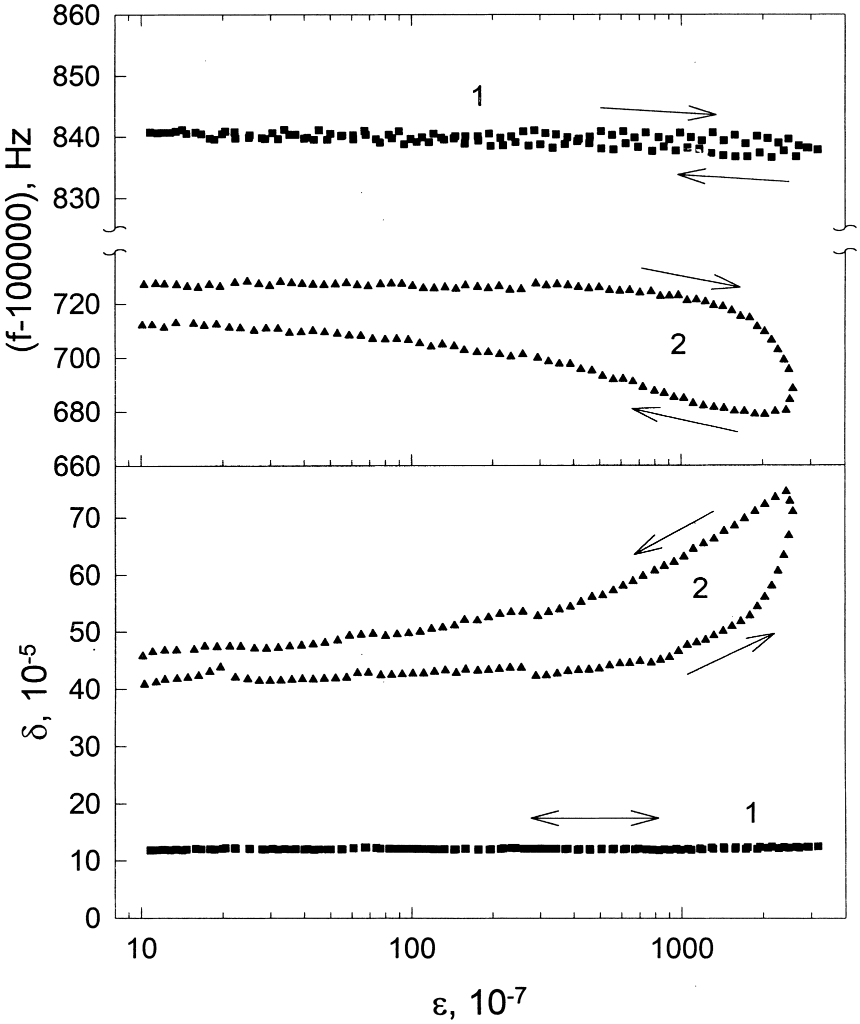

Fig. 4. Amplitude dependence of resonant frequency *f* and logarithmic decrement δ of neutron irradiated V–4Ti–4Cr alloy (produced in RF) before (1) and after (2) pre-straining. The arrows identify the direction of changes in amplitude ε. The sample was in liquid Li during irradiation (480°C).

3.3. V–10Ti–5Cr alloy

A wider program of acoustic tests has been carried out with this alloy composition. The tests have been performed with samples irradiated by protons and neutrons (Table 1). These samples from different batches were denoted by V11 and V2 in Table 1. Materials of these batches had a slightly different Cr content: V11 – 5%, V2 – 6%. The data of Table 1 give acoustic parameter changes for this material due to preliminary deformation. Note that only for the non-irradiated sample of V11, Young's modulus slightly decreases; the modulus of other samples increases. This indicates an elastic hardening of the material after deformation (notice that the plastic deflection of the non-irradiated samples and the proton irradiated samples was about 0.1 mm; for the neutron irradiated samples, there was almost no residual deflection, as well as for the V–5Ti–5Cr alloy). Nevertheless, one can find an anelastic softening effect as a result of both proton and neutron irradiation. This is clearly seen from the amplitude-dependent decrement δ_h and modulus defect $(\Delta E/E)_h$ behaviour by comparing sample No.9 with No.11, and 12 and sample No.10 with No.13 (Table 1). The sample No.14 irradiated in liquid Li reveals no radiation-induced softening when compared to the non-irradiated sample No.10.

It is interesting to notice a correlation in the behaviour of acoustic 'stress–anelastic strain' curves and macroscopic deformation diagrams [1]: in both cases, samples irradiated by protons were softer, than non-irradiated samples. Unfortunately, a similar comparison for neutron irradiated materials is not available.

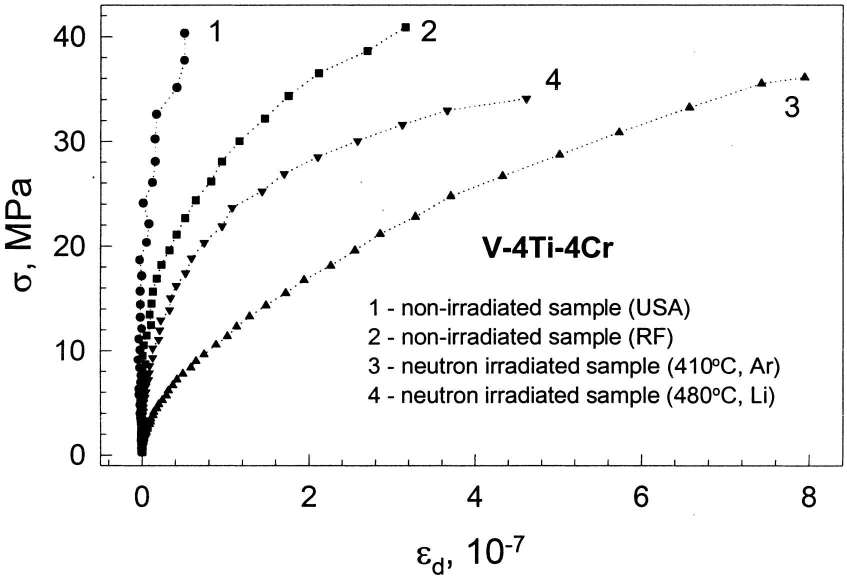

Fig. 5. Stress–anelastic strain curves obtained from acoustic data for non-irradiated (USA and RF) and neutron irradiated (RF) V–4Ti–4Cr samples under argon and in liquid Li environments. Non-linear anelastic strain $\varepsilon_d = \varepsilon(\Delta E/E)_h$; stress $\sigma = E\varepsilon$.

3.4. Undoped vanadium

As for the undoped vanadium, we had irradiated samples from different batches (V7 and V8), with neutrons and protons. They were slightly different as one can see from the acoustic properties of unirradiated samples, presented in Table 1. As to the irradiated samples, they show the elastic softening effect due to irradiation when comparing the data on amplitude-independent modulus defect $(\Delta E/E)_i$ for unirradiated and irradiated samples, and change only insignificantly their non-linear $(\Delta E/E)_h$ properties (Table 1). Note that the sample irradiated by neutrons (No.18 in Table 1), had a higher mass and dimensions (its cross-section was about 3·3 mm^2), as compared with the non-irradiated sample (No.16), and its preliminary deformation was made not by bending, as for other samples, but in a knife straining unit. For this reason, a quantitative comparison of the data is too difficult. One can only note a significant value of the Young's modulus defect $(\Delta E/E)_i$ measured at small amplitudes and a relatively small value of the amplitude-dependent modulus defect $(\Delta E/E)_h$, as for proton irradiated sample No.17.

4. Discussion

As was mentioned above, when studying the acoustic properties of vanadium alloys, two new effects have been revealed in this work. One of them is the elastic/anelastic softening effect of various alloys which arises due to neutron or proton irradiation, the second one is the anelastic hardening of some alloys after pre-deformation and irradiation. The hardening effect means an increase in measured effective elastic Young's modulus, and the softening means the modulus decrease. Both effects will be discussed below separately.

4.1. Neutron and proton softening effect

Fig. 6 summarizes microstrain properties of the alloys (RF production) studied in this work before and after neutron irradiation. One can see that, for the non-irradiated materials, an increase in the alloy content hardens the material by solid solution strengthening. Note that the V–4Ti–4Cr and V–5Ti–5Cr alloys show almost the same non-linear anelastic properties. An increase in the Ti-content makes the V–10Ti–5Cr alloy harder.

A softening effect under neutron (410–480°C) and proton (RT) irradiation was found in this work for some vanadium materials not only for the amplitude-independent damping range (this effect for V–10Ti–5Cr was first described in Ref. [1]), but for higher amplitudes, where anelastic deformation becomes non-linear. A champion in Young's modulus decrease for non-linear high amplitude range is the V–4Ti–4Cr alloy (Fig. 6 and Table 1) where $(\Delta E/E)_h = 0.22\%$. V–10Ti–5Cr softened in this amplitude range under neutron and proton irradiation as well, although not to this extent (curves 3 in Fig. 6). V–5Ti–5Cr samples irradiated by neutrons behave in another way: they show irradiation induced anelastic hardening (curves 2 in Fig. 6).

It is necessary to note that the behaviour of the non-irradiated V–5Ti–5Cr sample (RF production) after

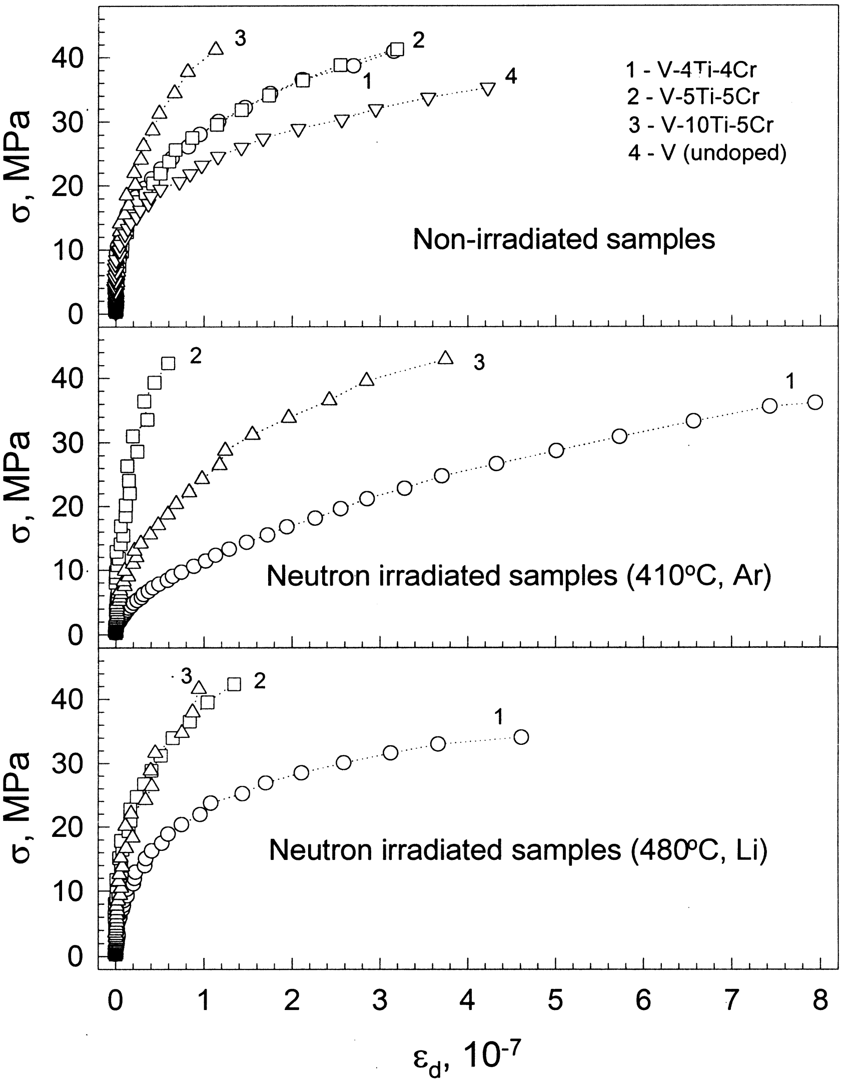

Fig. 6. Stress–anelastic strain curves obtained from acoustic data for non-irradiated and (in argon and liquid Li) neutron irradiated vanadium alloys produced in RF. Non-linear anelastic strain $\varepsilon_d = \varepsilon(\Delta E/E)_h$; stress $\sigma = E\varepsilon$.

deformation was qualitatively closer to that of undoped vanadium than to other alloys (Table 1). A reasonable explanation consists in a non-uniform distribution of alloying elements in the material, as well as in a different texture of the samples.

The highest softening effect after pre-deformation in the low amplitude range, where the materials behave linearly, was observed for neutron irradiated samples of undoped vanadium and V–4Ti–4Cr alloy (Table 1).

The elastic modulus softening effect in the amplitude-independent damping range (negative amplitude-independent modulus defect) after pre-straining is usually explained by fresh dislocations, which differ from aged ones [4]. The fresh dislocations are less pinned by point defects (e.g. Cottrell clouds). So, they are able to move easily and to add higher anelastic dislocation strain to the elastic one under vibrational load. The fact that as a result of irradiation we observe a noticeable increase of an elastic deformation (both linear and non-linear) for some alloys, can be explained by either a decrease of the barriers concentration for moving dislocations or the appearance of centres, which make the movement of dislocations easier. The first explanation is of low probability. The second one needs the creation of some 'lubrication' agents due to irradiation. Vacancies were named in Ref. [5] as the most probable entities to explain the softening effect which was observed for aluminium under electron irradiation at a low (5.7 K) temperature. It is quite probable, that (at room temperature) vacancies can play the same role in vanadium alloys as well although the data for the V–5Ti–5Cr alloy look very strange in this explanation. This alloy hardens after irradiation according to the data of non-linear anelastic strain (see Fig. 6). It is also difficult to explain the data on linear anelastic strain (modulus defect $(\Delta E/E)_i$, Table 1), where a transition is clearly seen for irradiated samples from a strong softening after deformation for undoped vanadium and the V–4Ti–4Cr alloy to a noticeable hardening of the V–10Ti–5Cr alloy: $(\Delta E/E)_i$ turns from large negative to large positive values. Evidently, besides lubrication agents, there should exist in V and V-alloys radiation induced hardening structure entities.

4.2. On increase of elastic modulus due to pre-straining

In Ref. [1], performed with V–10Ti–5Cr, the effect of Young's modulus increase has been found resulting from preliminary deformation of a sample irradiated by protons. The elastic hardening was discussed due to a pinning of dislocations by segregated hydrogen atoms. The experimental data of this work indicate that this is more likely not the case: positive values of $(\Delta E/E)_i$ (Table 1) are found for non-irradiated samples of all alloys and for neutron-irradiated samples of the V–10Ti–5Cr alloy. Moreover, irradiation of undoped vanadium with a high proton fluence leads not to hardening, but to elastic softening of the material. The analysis of the $(\Delta E/E)_i$ data in Table 1 leads to the conclusion that this value depends mainly on the alloy composition and preparation technology. Structure analysis has shown that American alloys (high positive $(\Delta E/E)_i$ values) consist of larger grains with a developed dislocation structure (high dislocation density) as compared to Russian ones. Irradiation is also very important, but there is no qualitative difference between neutron and proton irradiations: the V–10Ti–5Cr alloy in both cases hardens and undoped vanadium softens.

An explanation for hardening that leads to positive values of the $(\Delta E/E)_i$ modulus defect after pre-straining, may be the anharmonicity of atom oscillations in the crystalline lattice (effect of higher order elastic constants). Higher values of resonance frequency reveal as a result of an increase of residual stresses in the sample after preliminary deformation. The anharmonicity of dislocation vibrational movement can also influence the effect.

Note that the increase of effective elastic modulus during quasistatic deformation was observed for a number of materials: Cu, W, Nb, NaCl, NaF, Si, Al [6]. However, an increase of the modulus for pre-strained samples has not been observed up to now. Probably, vanadium alloys are the first for which this effect has been found. The analysis in Ref. [6] has shown that the elastic modulus increase under static load is, most probably, due to either lattice or dislocation anharmonicity (see also Ref. [7]). It is quite possible that residual stresses in the samples of vanadium alloys can be higher after preliminary plastic bending. Note that irradiation by both neutrons and protons leads to a noticeable relaxation of internal stresses for vanadium and V-alloys (except V–5Ti–5Cr) in the framework of this explanation.

The results obtained in this work might reveal another mechanism of dynamic elastic modulus changing. This mechanism, together with dislocation vibrational movement (this effect always reduces the effective modulus) can exist in principle of both signs due to elastic constants of higher orders (anharmonicity of atom oscillations in lattice). The modulus defect arises here as a result of change (increase or relaxation) of internal stresses. Internal stresses can relax also due to high amplitude vibrations; so, this mechanism can influence, the value of the amplitude-dependent modulus defect $(\Delta E/E)_h$.

4.3. Amplitude hysteresis. Cross-over effect

High amplitude ultrasonic excitation usually leads to an amplitude-dependent damping and negative elastic modulus defect, or their noticeable increase [3]. Vanadium and V-alloys, before and after irradiation by protons and neutrons, show the same behaviour. At room temperature, an amplitude hysteresis is observed in these materials: the curves do not coincide when increasing and then decreasing the amplitude. The decrement usually increases and the resonance frequency (elastic modulus) decreases after measurements at high amplitudes. However, this is not the case for the decrement of the neutron irradiated V–4Ti–4Cr sample after pre-straining (Fig. 3, curve 2) for which a cross-over effect is clearly seen.

The non-linear anelastic strain together with the non-linear amplitude-dependent damping and elastic modulus defect is usually ascribed to vibrating dislocations which overcome point defects stress fields. The amplitude hysteresis appears in this model as a result of pinning points redistribution under rather high vibrational stresses. These changes can be due to either point defects diffusion or change of the coupling force between the point defect and dislocation after depinning (it may become zero as well). In this way, the amplitude-dependent and amplitude-independent decrements and modulus defects redistributions may take place. New relationship δ_h/δ_i can explain a cross-over effect a similar phenomenon has been found experimentally for ceramic high-temperature superconductors Y–Ba–Cu–O at liquid helium temperatures [8].

An evolution of the 'dislocation–point defects' structure under the influence of vibrational stresses is one of possible mechanisms, leading to the relaxation of internal stresses. Reduced internal stresses can decrease the resonance frequency of a sample due to, for example, lattice anharmonicity. A contribution of this mechanism might be conform with the following observations: (1) the effect of increase in the measured elastic modulus after pre-bending of the vanadium alloys and, (2) the evidence that the amplitude-dependent modulus defect $(\Delta E/E)_h$ for these materials was essentially higher (in absolute value) than the amplitude-dependent decrement: for the majority of samples, studied in this work, the ratio $\delta_h/(\Delta E/E)_h < 1$ (Table 1). Note that this ratio is higher than one for other (Cu and its alloys, Al, Zn, NaF, MgO) materials [9]. An additional contribution to non-linear deformation can evidently arise and this is not accompanied by a noticeable increase in dissipated (absorbed) energy of elastic wave.

5. Conclusion

The experimental data obtained for vanadium and its alloys have shown that the acoustic methodology gives a unique possibility to study the initial stage of plastic deformation (at the small level of 10^{-8}–10^{-6}) when the dislocation movement in the material is still reversible. It has been shown that composition, producing technology, irradiation by protons and neutrons, and, possible, irradiation environment have a pronounced effect on elastic/anelastic properties of the vanadium alloys.

A brief qualitative consideration of the results allowed us to discuss possible mechanisms which control the elastic/anelastic properties of V and V-alloys studied in acoustic experiments.

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