

# Structure and strength characteristics of dispersion-hardened composite foils (films) based on iron and nickel

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Effects of hardening components (W, SiO, Al<sub>2</sub>O<sub>3</sub>, and ZrO<sub>2</sub>) on structure and physical-mechanical characteristics of Fe and Ni composites were studied. The content of the hardening phases was up to 2 vol.%. It was shown experimentally that physical-mechanical characteristics of the prepared nano-crystalline materials were defined by the structure and inhomogeneous distribution of the hardening phase within a grain. The reasons for high thermal stability of the nano-composites were established. The factors determining the nano-composite relaxation resistibility and resources for its increasing were revealed.

В работе изучалось влияние упрочняющих элементов (W, SiO, Al<sub>2</sub>O<sub>3</sub> и ZrO<sub>2</sub>) на структуру и физико-механические характеристики композитов Fe и Ni. Содержание упрочняющих фаз составляло до 2 об. %. Экспериментально показано, что физико-механические характеристики полученных нанокристаллических материалов определяются структурой и неоднородностью распределения упрочняющей фазы в пределах зерна. Определены причины высокой термической стабильности нанокompозитов. Выявлены факторы, определяющие релаксационную стойкость нанокompозитов и пути ее повышения.

*Структура і міцнісні характеристики фольг (плівок) дисперсно-зміцнених нанокompозитів на основі заліза і нікелю. О.І.Ільїнський, О.Є.Бармін, С.І.Лябук.*

У роботі вивчався вплив зміцнюючих елементів (W, SiO, Al<sub>2</sub>O<sub>3</sub> і ZrO<sub>2</sub>) на структуру і фізико-механічні характеристики композитів Fe і Ni. Зміст зміцнюючої фази становило до 2 об. %. Експериментально показано, що фізико-механічні характеристики отриманих нанокристалічних матеріалів, визначаються структурою і неоднорідністю розподілу зміцнюючої фази в межах зерна. Визначено причини високої термічної стабільності нанокompозитів. Виявлено фактори, що визначають релаксаційну стійкість нанокompозитів та шляхи її підвищення.

## 1. Introduction

Interest in sub-micro- and nano-crystalline (smc and nc) materials with unique physical-mechanical properties increases headily. The precursor of nano-materials science era is Nobel Prize winner R.Feinman, who in 1959 put forward an idea to construct small-sized objects on atomic level [1]. Later on, in 1980<sup>th</sup> G.Gleiter formulated the main concepts for smc and nc materials [2].

However, as long ago as in 1960<sup>th</sup> the works appeared on studying vacuum deposited thin metallic films with high disperse (as a matter of fact, nano-crystalline) structure and unusually high strength level [3–9]. The integration approach to the problem in subsequent works of L.S. Palatnik's school allowed foundation of film physical materials science. The summarizing monograph [10] was based on two fundamental theses according to which film structure and properties are determined by size effects and

non-equilibrium states. These these are considered as the base ones for studying modern nano-materials [11–14].

One of the promising ways to conserve the thin film state in objects with thickness of the order of 0.1 mm was found to be creation of dispersion-hardened composites with refractory nano-particles of strengthening phase. With alloying element content only ~ 1 vol.%, the nano-composites were remarkable for very high and stable strength [15].

Usually it is thought [13–14] that compact nano-materials, to which thin films are concerned, will be the most applicable as construction and functional materials for new technologies. In this connection, their mechanical properties deserve special attention.

An important little investigated characteristic of films (foils) is also their relaxation resistibility that is of special significance for non-equilibrium material in electronics, precision engineering and other fields of the new technique. Quality and longevity of instruments is limited by material size non-stability because of relaxation process development under stress, temperatures and other factors [16–18].

Below new results of studying the structure and physical-mechanical characteristics ( $\sigma_{0.2}$ , HV,  $\rho/\rho_0$ , and  $\Delta\sigma/\sigma_0$ ) of dispersion-hardened nano-composite foils (films) prepared from the most important construction materials — iron and nickel are presented. The main aim is analysis of size effects, thermal stability and relaxation resistibility of the objects.

## 2. Experimental

Foils (films) of about  $\leq 30 \mu\text{m}$  thickness were prepared by simultaneous electron-beam evaporation of main metals (Fe and Ni) and hardening elements and subsequent deposition onto glassceramics substrates. For Fe films, it was tungsten; for Ni films —  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  oxides. Content of the hardening phases did not exceed 2 vol.% and was controlled by X-ray spectral and element mass analyses. Diffractometer DRON-2 and transmitting electron microscope PEM-200 were used for the investigations. Deformation experiments in the regime of active extension followed by stress relaxation were carried out using the instrument TIRAtest-2300 with high rigidity. The stress relaxation in the foils was estimated by the ratio  $\Delta\sigma/\sigma_0$ , where  $\Delta\sigma$  is the relaxation value for 0.5 hours after

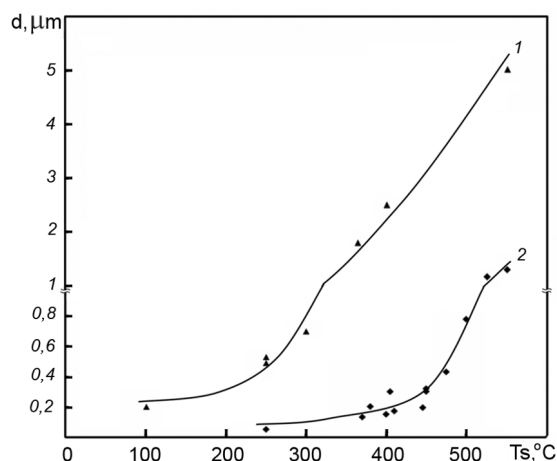


Fig. 1. Substrate temperature effect onto the grain average size of vacuum condensates: 1 — Fe; 2 — Fe-W (W ~ 1.1 vol. %).

stopping the active extension;  $\sigma_0$  is the initial stress value close to yield point  $\sigma_{0.1}$ . Note that the relative depth of relaxation  $\Delta\sigma/\sigma_0$  is the value reciprocal to the relaxation resistibility.

## 3. Results and discussion

### 3.1. Size effects

It is known that one of the ways to increase strength and thermal stability of non-equilibrium metallic materials is alloying with admixtures of small concentration. In the case of foils (films) crystallized from the vapour phase in vacuum, such alloying is realized by simultaneous evaporation of constituents [15, 19, 20]. At that, the alloying element besides refractivity and high elastic modulus should be practically insoluble in the metallic matrix.

Note that such approach is successfully realized for preparation of the modern nc and smc materials [12–14].

Let us consider the results of studying the vacuum condensates of the most important constructional material — iron. The alloying element was tungsten satisfying the requirements above.

The main parameter determining the degree of structure dispersity is the substrate temperature  $T_s$  under crystallization (Fig. 1). As it is seen, even micro-alloying by tungsten results in significant dispersion of the structure in the whole range of crystallization temperatures and allows decreasing the average grain size of iron based to nano-level ( $d \approx 50 \text{ nm}$ ). The effect of alloying

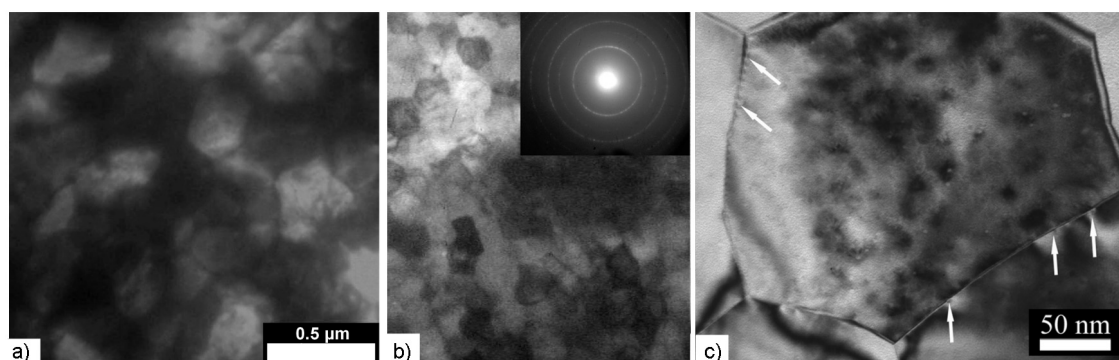


Fig. 2. Structure of vacuum condensates Fe (a) and Fe-W (b, c) in initial state.

increases as the Fe atom diffusion mobility rises with increasing temperature  $T_s$ .

For Fe-W films, single phase structure is typical that is supposed by electron-diffraction and X-ray diffraction patterns. However, qualitative analysis of bright-field images at high magnification indicates the presence of contrast in grain boundary areas of Fe matrix. The contrast may be interpreted as segregation of W nano-particles of several nano-meters size (in Fig. 2 these are pointed by arrows).

X-ray diffraction measurements of Fe-W composite did not show any changes of the crystalline lattice parameter. This means that high degrees of supercooling and supersaturating under crystallization do not result in formation of supersaturated solid solution of tungsten in iron. The fact is connected, seemingly, with pushing aside the alloying element by the crystallization front, and as a result, with formation of W segregations at the growing Fe nuclei boundaries.

Besides the crystallization temperature, also W content influences significantly on the film grain structure (Fig. 3). The greatest dispersion of the structure takes place under increasing W content up to  $\sim 0.3$  vol.%, while under the further W concentration increase, the grain size is practically not changed. Such character of the dependence remains for all deposition temperatures, the dispersion degree increasing with decreasing the diffusion mobility of vacuum condensate atoms.

The non-equilibrium degree of Fe-W nano-composites determines the level of mechanical characteristics thereof (Fig. 4). The largest values of micro-hardness and yield stress are 3.3 and 1.2 GPa, respectively. Thus, the empirical relation  $HV \approx 3\sigma_{0.2}$  typical for nc materials is applied [12–14]. Note that under decreasing  $T_s$  to 250°C and dispersing

the structure to nano-level ( $\sim 50$  nm) the film hardness reaches 5.5 GPa. However, because of their low ductility, extension experiments are complicated.

Conventionally, to describe the dependence

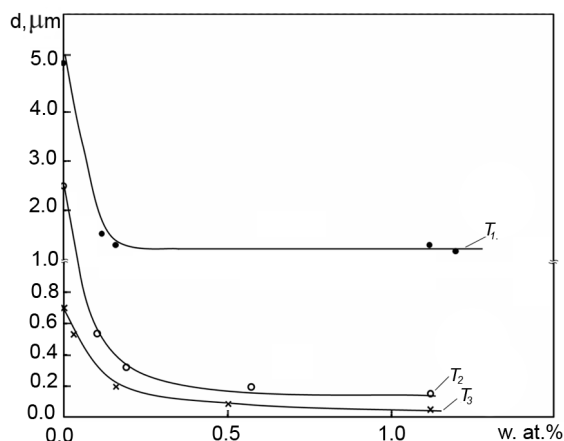


Fig. 3. Influence of tungsten content onto the grain average size of iron vacuum condensates: 1 —  $T_s = 550^\circ\text{C}$ ; 2 —  $T_s = 450^\circ\text{C}$ ; 3 —  $T_s = 250^\circ\text{C}$ .

of strength properties on the grain size, Hall-Petch relation is used  $\sigma_{0.2} = \sigma_0 + kd^{-1/2}$ , where  $\sigma_0$  is a physical yield stress for single crystals, and  $k$  characterizes the hardening degree [21]. As it is seen from Fig. 5, for Fe and Fe-W foils (films) the Hall-Petch law is fulfilled in the whole studied range of grain sizes.

At that, the  $k$  parameter for Fe foils is close to corresponding values for armco-Fe of metallurgical industry that formally indicated similarity of hardening mechanisms for vacuum deposited and metallurgical materials.

As to noticeable increase of the  $k$  parameter for Fe-W composite with grain average size larger than 350 nm, such the effect was repeatedly observed in metallic nc-

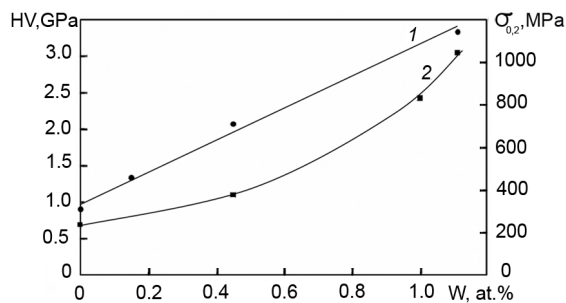


Fig. 4. Influence of tungsten content onto mechanical characteristics of iron vacuum condensates: 1 — micro-hardness (HV); 2 — arbitrary yield stress ( $\sigma_{0.2}$ ).

materials [12–14], and in our case it may be connected with W segregation at the grain boundaries. However, at  $d$  values less than  $\sim 350$  nm, the tendency to changing (decreasing)  $k$  parameter is observed, which is also typical to many nc materials [12–14].

As the problem is of special interest, we notice only that the observed effect is usually connected with increasing role of the grain boundary sliding under significant dispersion of the structure.

Interesting is to compare the obtained results with the known ones for Fe based nc materials. For example, in the steel 3 treated by intense plastic deformation the value  $\sigma_{0.2}$  achieves 0.98 GPa [11]. In addition, the strength characteristic level in Fe–W films ( $\sigma_{0.2} \approx 1.2$  GPa) realized at 1.1 at % W is achieved in technical alloys only at W content larger by a factor  $\sim 20$  (20 vol % W).

### 3.2. Thermal stability of nano-composites

Thermal stability of functional materials is the most important and intensively studied problem. The peculiarities of nc and smc structures predetermine extra high degree of non-equilibrium of the objects under investigation, in which under thermal and force acting, the processes of homogenization, segregation, recrystallization, and relaxation develop resulting in structure evolution, subsequently, physical and mechanical properties [14, 22].

In this connection, we consider the results obtained for Fe–W films [23–25]. Along with strength characteristics also the relative electrical resistivity was measured. Investigations showed that the grain average size (about 500 nm) remains unchanged in the time gap studied up to 800°C, however,

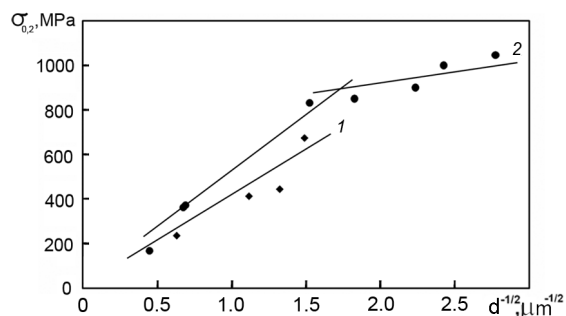


Fig. 5. Dependence of arbitrary yield stress ( $\sigma_{0.2}$ ) on the foil grain average size: 1 — Fe; 2 — Fe–W (W  $\sim 1.1$  vol. %).

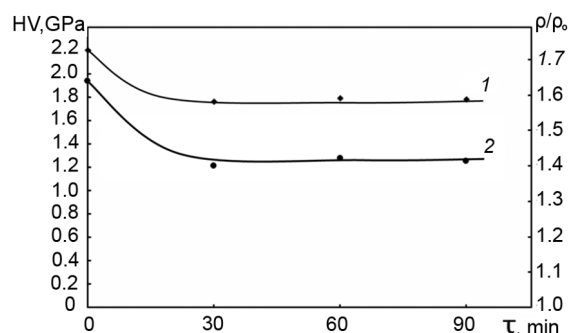


Fig. 6. Effect of isothermal annealing time (800°C) on the physical and mechanical characteristics of F–W vacuum condensates: 1 — micro-hardness (HV); 2 — relative electrical resistivity ( $\rho/\rho_0$ ).

the values HV and  $\rho/\rho_0$  decrease significantly after annealing for 30 min (Fig. 6).

As it was mentioned in [14], the electro-resistivity vs. temperature dependence of compact nc materials is used for analysis of grain boundary state and determination of their relaxation temperature. The resistivity is described by the following relation:

$$\ln\left(\frac{\rho}{\rho_0}\right) = \frac{l_0}{d} \ln\left(\frac{1}{r}\right), \quad (1)$$

where  $\rho$ ,  $\rho_0$  are resistivity of poly- and single-crystalline film respectively;  $l_0$  is the average free path of an electron in the defect-free single-crystal;  $d$  is the grain average size;  $r$  is the electron scattering coefficient when crossing the grain boundary.

As  $0 < r < 1$ , it follows from the relation given that the resistivity of nc materials should increase with  $d$  decreasing. Indeed, significant increase of the resistivity was mentioned by many researchers [12–14], and also is observed for our objects (Fig. 6). The revealed fact of significant decreasing  $\rho/\rho_0$  under annealing the Fe–W films at un-

Table 1. Local element composition of Fe–W films

Element	Total spectrum	Spectrum 1	Spectrum 2	Spectrum 3	Spectrum 4
Fe, vol. %	99.81	99.96	99.79	99.17	99.65
W, vol. %	0.19	0.04	0.21	0.83	0.35

changed grain size indicates variation of the scattering coefficient  $r$  and, consequently, decreasing equilibrium degree of the grain boundaries. This effect may be connected with removal of internal stress, healing the micro-pores and grain boundary discontinuities, etc. [12–14]. It is considered that such structure relaxation precedes the grain growth.

Actually, at temperatures higher than 800°C in the Fe–W films the recrystallization processes develop resulting in substantial decreasing physical-mechanical characteristics HV and  $\rho/\rho_0$ . It is worth to note that in commercial Fe–W alloys such processes take place at temperatures by 300°C as lower.

Thus, the Fe–W films containing only about 1.1 vol. % W possess rather high strength characteristics and high thermal stability that is the most important for the smc and nc structures. As it was mentioned above, these features of the nano-composites may be related with formation of W segregations at the grain boundaries and sub-boundary areas.

For supporting the fact, the EDS technique was applied to carry out the local element analysis (Fig. 7). Comparative measurements were done in the bulk of the grain (spectrum 1), triple joint of boundaries (spectrum 2), and at boundaries and sub-boundary zones (spectra 3 and 4). The data are given in Table 1.

As it is seen, the data clearly indicate formation of segregations because W content at the grain boundaries and sub-boundary areas is by a factor 5 to 20 higher than the values in the grain bulk.

### 3.3. Relaxation resistibility of nano-composites

As it was mentioned above, one of the features of the smc and nc structure evolution is the structure relaxation connected with transition into the more equilibrium state. On the other hand, the most important deformation characteristic of the materials is their relaxation resistibility related with external stress relaxation.

Below the analysis of the structure influence onto the regularities of deforming stress relaxation in Ni-based nano-compos-

ites is presented which determines their relaxation resistibility.

The Ni-based composites have the smc structure with 200–500 nm size grains separated by high-angle boundaries (Fig. 8). Oxides as nano-disperse particles of 5–10 nm are rather uniformly distributed over the metallic matrix and are revealed as local elements of contrast. Electron-diffraction patterns indicate amorphous structure of the particles. Absence of preferential grain boundary segregation of the hardened phase revealed in the Fe–W films is connected evidently with lower diffusion mobility of oxide nano-particles in the metallic matrix and "immuring" them in the bulk of growing grains [20].

According to the X-ray diffraction data, insertion of the oxide particles results in no noticeable variation of Ni crystalline lattice parameter. This indicates absence of any substantial diffusion interaction between the oxides and the matrix metal.

Alloying by oxides results in strong hardening of the nickel films; the most effect is observed under increasing the oxide content up to ~ 1 vol. %. At the oxide concentration more than 1 vol. %, insignificant hardening is followed by brittleness evolution. The typical results are given in Table 2. Note, that the largest values of micro-hardness at 1.5–2.0 vol. % of oxide content are about 4 GPa.

Let us carry out comparative estimation of the obtained results. For example, the yield stress of the best age-hardened composites Ni–ThO<sub>2</sub> (2.5 vol. %) is 600 MPa [26]. Additionally, the nano-crystalline Ni with grain size of 15 nm has the yield stress 940 MPa [12]. Thus, film composites containing only 0.5 vol. % of oxides are comparable by strength level with the nano-crystalline nickel and substantially surpass the last by thermal stability. Let us consider the reasons of the film composite high strength level. It is known that both the hardening phase particles and the grain high-angle boundaries are the most effective barriers for moving dislocations. The barrier effect increases with decreasing both the particle size and distances between them, as well as with the grain dispersing.

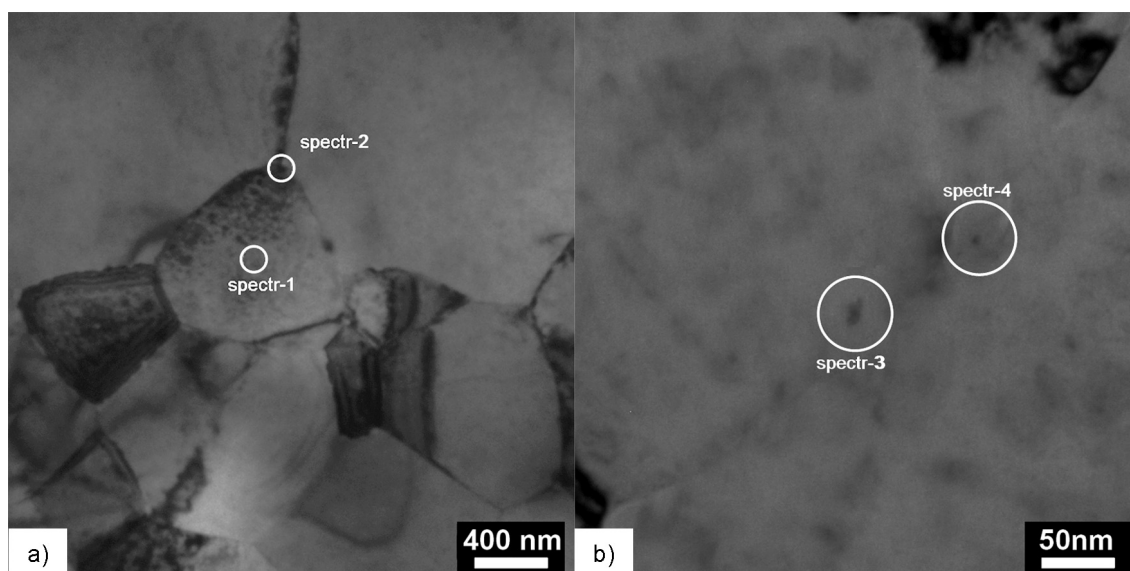


Fig. 7. Results of point EDS element analysis carried out for Fe-W foils ( $T_s = 550^\circ\text{C}$ , W ~ 0.2 vol. %).

Therefore, the very high level of nano-composite strength is caused by combined influence of the grain-boundary hardening and dispersion one. The observed difference of  $\sigma_0$  values for the composites (Table 2) is connected mainly with different dispersion of the oxides [26]. Analysis of stress relaxation showed that for all studied composites, non-monotonic variation of the relaxation relative depth  $\Delta\sigma/\sigma_0$  with oxide concentration ( $f$ ) is observed (Fig. 9).

So, as  $f$  values increase to about 1 %, the hardening is followed by  $\Delta\sigma/\sigma_0$  increasing, and, subsequently, decreasing the relaxation resistibility of the composites. Further content increase, along with remaining increase of the yield stress, is followed by  $\Delta\sigma/\sigma_0$  drastic decreasing and, hence, raising the relaxation resistibility. To analyze the new effect of the relaxation resistibility non-monotonic changing we consider influence of the hardening phase onto the composite structure. As it is known, the main structure parameters influencing on the stress relaxation are the grain sizes of metallic matrix and hardening oxides. The oxide dispersion is practically unchanged in the concentration range under study. However, the grain sizes decrease by a factor 3–4 as  $f$  increases to 0.2–0.3 vol. %, and

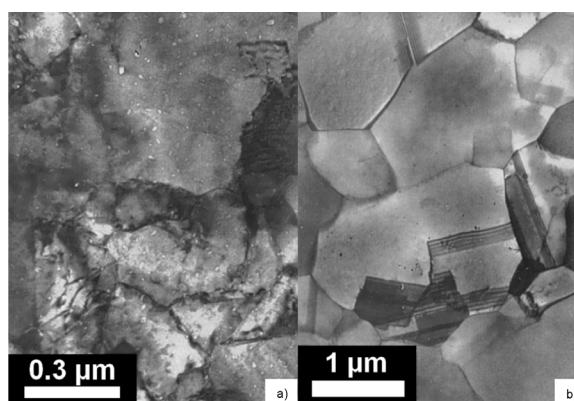


Fig. 8. Structure of vacuum condensates: a — Ni-SiO; b — Ni.

are practically unchanged under further concentration increase. Our data indicate substantial influence of the grain dispersion on the relaxation resistibility. So, for homogeneous Ni foils (films) under decreasing the grain size from 5 to 1  $\mu\text{m}$ , the value  $\Delta\sigma/\sigma_0$  increases by a factor of 4–5. This effect may be connected with increasing density of mobile dislocations formed under stress at the grain high-angle boundaries. Naturally, the density of grain boundaries increases as the grain size decreases. As to the particles of the hardening phase, as it is known,

Table 2. Comparative strength values for composites with oxide content 0.4–0.6 vol. %

Material	Ni foils (commercial purity)	Ni films (vacuum)	Ni-SiO (vacuum)	Ni-Al <sub>2</sub> O <sub>3</sub> (vacuum)	Ni-ZrO <sub>2</sub> (vacuum)
Yield stress, $\sigma_0$ , MPa	100	260	600–650	700–800	850–1100

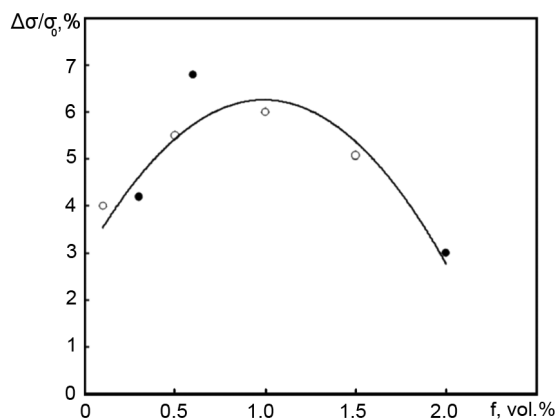


Fig. 9. Composition effect on the relative relaxation depth for composites: ● — Ni-SiO; ○ — Ni-Al<sub>2</sub>O<sub>3</sub>.

these are stress local sources [27]. The level of the stress depends on differences between elastic modules, thermal extension coefficients of the particles, etc. The residual stress at phase boundaries is able to stimulate local plastic deformation under applied external stress. As the stress relaxation is determined by density of mobile dislocations  $\rho$ , and their free path  $\lambda$ , we get  $\Delta\sigma = \rho b \lambda$ , where  $b$  is the Burgers vector,  $\lambda$  is an average distance between oxide particles in the slip plane [28]. Thus, as oxide content increases, the number of nano-phase barriers (particles) increases progressively and, respectively, the  $\lambda$  value decreases. On the other hand, the particles appearing may be sources of mobile dislocations, which density depends on the volume fraction of the particles. Then, as it was mentioned above, when the particle content decreases to 0.2–0.3 vol. %, sudden dispersing the grain structure occurs and, hence, growing the density of mobile dislocations at high-angle crystallite boundaries. Consequently, the observable increasing  $\Delta\sigma/\sigma_0$  with oxide content increase is determined by mobile dislocation active sources which are both crystallite and phase boundaries. Changing the dependence at the oxide content more 1 vol. % may be connected with strengthening interaction of stress fields due to progressive shortening distances between particles as their concentration increases. Estimation of corresponding average critical distance between particles gives a value about 80 nm, which generally depends on the oxide dispersion. Note, that a certain contribution into  $\Delta\sigma/\sigma_0$  values also may be included by variously scaled defects of a crystalline lat-

tice (growth dislocations, twins, nano- and micro-pores, etc.).

Thus, the revealed effect of  $\Delta\sigma/\sigma_0$  parameter non-monotonic variation can be considered as a consequence of competitive influence of hardening oxide particles which are simultaneously sources and stoppers of the mobile dislocations.

Earlier, for dispersion-hardened films of the nano-composites it was shown that besides strengthening under active extension these demonstrate also strengthening under relaxation of deforming stress [29]. The evolution of ideas on this important effect allowed proposition of the way to increase relaxation resistibility of such high durable objects. The method is in realizing the repeating (cycling) relaxations at the stress level about  $\Delta\sigma_{0.1}$  which does not result in decreasing the composite plasticity. The corresponding treatment changes, in principal, the character of the relaxation resistibility dependence on the oxide concentration. So, in whole concentration range the value  $\Delta\sigma/\sigma_0$  decreases by a factor of 3–4, that means the corresponding increase of the relaxation resistibility. This effect may be connected with exhausting the sources of the mobile dislocations at the grain and phase boundaries as well as blocking the dislocations by the oxide qnano-particles. Healing the nano- and micro-pores under loading also may give a certain contribution. Note that after triple repeated relaxations, significant growth of the yield stress is observed (~ 20 %). This method can be considered as a special case of the known program hardening the non-equilibrium bulk metallic materials.

Thus, the cycling relaxation of deforming stress allows obtaining the objects combining high values of durability and relaxation resistibility. The fact has a principal significance for practical application of the nano-composite films.

#### 4. Conclusions

The possibility of nano-structure formation in vacuum deposited dispersion-hardened foils (films) was shown. The grain minimum size in Fe films containing only 1.1 vol. % W is 50 nm. It was established that the Hall-Petch dependence for Fe and Fe-W foils is fulfilled in whole investigated range of the grain sizes; however, for Fe-W composite with the grain average size lower ~ 350 nm, the  $k$  parameter decrease typical for the nc materials was observed. The segregation of alloying W at the grain boundaries was found, which stimulates increasing

strength characteristics and thermal stability of the nano-composites. The results obtained indicate the possibility to realize nano-crystalline state in dispersion-hardened foils with thicknesses about 30  $\mu\text{m}$ .

The factors influencing on the nano-composite relaxation resistibility were revealed. It was shown that the cycling relaxation of deforming stress permits obtaining the materials combining high durability and relaxation resistibility values.

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