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Relaxation Structure Formation in Deformation of Nickel

E. Zasimchuk, Yu. Gordienko, L. Markashova, and T. Turchak

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The purpose of this study was to quantitatively investigate the relaxation structure formation in polycrystalline nickel deformed by rolling at room temperature. The relaxation structures were examined on polycrystalline nickel with a purity of 99.99% Ni, deformed by rolling. The sub-structure of nickel after rolling to 50-80% was examined by the TEM and X-ray analysis methods by line profile analysis. It was revealed the presence of three qualitatively different types of structural elements: microbands of a macroscopic length; cells of different shapes and dimensions; micrograins less than 80 nm in size. The normalized curves of distribution of cells dimensions coincide, which suggests a scale invariance of these parameters. On the other hand, addition of normalized dimensions of micrograins to statistical dependence of dimensions of the cells leads to distortion of the dependence. The mechanisms of formation of the cells and micrograins to be different: micrograins were shown are nuclei of primary recrystallization.

Keywords aluminum, electron microscopy, micrograins, primary metals, recrystallization, rolling, scaling

Formation of structure in metal during and after plastic deformation may occur according to two scenarios:

- *relaxation structure formation* (formation of cellular and polygonal structure, recrystallization), which is characterized by the kinetics that depends on the temperature, nature of a material, and deformation conditions; scaling (scale invariance) of dimensions and disorientations (Ref 1-10);
- *self-organization of deformed crystal, i.e. synergetic structure formation*, which is characterized by the stepwise kinetics (can be described using the δ-function), cooperative character of the process that simultaneously involves macro volumes of the material being analysed, and fractal morphology of different scales of structures (Ref 11-13).

The purpose of this study was to quantitatively investigate the relaxation structure formation in polycrystalline nickel deformed by rolling at room temperature. The relaxation structures were classified through their statistical examination by the method of transmission electron microscopy (TEM). The special attention was given to the mechanism of formation of different types of the structures, which was studied by comparing the normalized curves of distribution of their dimensions.

The relaxation structures were examined on polycrystalline nickel with a purity of 99.99% Ni, deformed by rolling. The sub-structure of nickel after rolling to 50-80% was examined by the TEM and X-ray analysis methods. The plastically deformed metal is a non-equilibrium system. This shows up, first of all, in the presence of micro and macrostresses, which can be easily determined by the X-ray method, e.g., by line profile analysis (Ref 14, 15). For analysis we used two pairs of lines: (111) and (222); (200) and (400). To illustrate, Table 1 gives values of microstresses (σ, MPa, $\times 10^4$) and dimensions of regions of coherent scattering (L, μm) for nickel deformed by rolling and tension. The line profile analysis method was used for the calculations. Figure 1 illustrates examples of profiles of X-Ray peaks (111) and (222) for nickel deformed by rolling to 50-80%.

It can be seen from the Table 1 that parameter σ first dramatically grows, and then decreases, reaching minimum in the fractured state (in tension). Parameter L, on the contrary, first decreases, and then grows up to fracture (in tension). Parameters σ and L in rolled nickel behave in the similar way, but the fracture during rolling is absent. This is indicative of intensification of the relaxation processes with increase in ε%. But it cannot be unambiguously stated that these processes accompany deformation, as X-ray analysis was conducted after unloading. At the same time, it is apparent that this behavior of the parameters determined by the X-ray method should be related to the relaxation structure formation, the result of which, i.e., sub-structure of nickel after rolling to 50-80%, was examined by the TEM method. Examples of the TEM structure of nickel (with purity of 99.99% Ni) are shown in Fig. 2 and 3.

Of notice is the presence of three qualitatively different types of structural elements (see Fig. 2 and 3):

(1) microbands of a macroscopic length;
(2) cells of different shapes and dimensions with blurred boundaries and variable internal contrast;
(3) micrograins with clearly defined boundaries and constant internal contrast (less than 80 nm in size).

The cells and micrograins are located both inside and outside the microbands. It can be noticed the micrograins are observed in our study for the first time.

One can be seen on the SAD patterns splitting of the diffraction reflexes. It may be connected with the advent of lump disoriented micrograins inside and outside the microbands.

E. Zasimchuk, Yu. Gordienko and **T. Turchak**, G.V. Kurdyumov Institute for Metal Physics of NAS of Ukraine, Kyiv, Ukraine; and **L. Markashova**, E.O. Paton Electric Welding Institute of NAS of Ukraine, Kyiv, Ukraine. Contact e-mail: eezas@imp.kiev.ua.

Comparison of Fig. 2 and 3 shows that increase in the deformation degree makes the cellular and micro-granular structure more pronounced, although the traces of microbands can be seen in some regions of foil. We carried out quantitative processing of structural elements of rolled nickel by using not less than 30 fields of vision for each deformation degree. It allowed the observation of every structural element more than 30000 times. We determined the dimensions of the cells and micrograins in two mutually perpendicular directions, as in the majority of cases these structural elements were not equiaxed.

As can be easily seen, to see many questions about deformation structures remain incompletely answered despite the sophistication of modern measurement and modeling capabilities. One of such questions concerns the application of universal scaling principle to deformation structure formation. Experimental data achieved the distributions of cells spacing and misorientation for different materials and deformation conditions (Ref 1-5) to fall on the same curve when normalized with the average value. This result attracted attention of a number of authors in the last few years (Ref 6-8). The similar pattern was revealed in our works for recrystallized grain growth in aluminum single crystal (Ref 9) and mechanical properties in a number of heat-resistant steels (Ref 10).

Figure 4 shows the normalized curves of distribution of the rolled nickel cells in dimensions. Statistical processing was carried out as described in studies (Ref 1-5).

As reported in studies (Ref 1-5), the normalized curves of distribution of disorientations and dimensions of the cells in a number of metals deformed under different conditions coincide, which suggests a scale invariance of these parameters. Our results shown in Fig. 4 allow a similar conclusion to be made for the cells of nickel rolled to different deformation degrees.

Consider now whether it is possible to combine the range of micrograins according to TEM for nickel rolled to 50-80% with the dependence shown in Fig. 4. The normalized curves of distribution in dimensions of cells and micrograins for the states of nickel in this study are combined in Fig. 5.

As can be concluded from Fig. 5, addition of normalized dimensions of micrograins to statistical dependence of dimensions of the cells (Fig. 4) leads to distortion of the dependence: the experimental points for micrograins in large part do not pack up on the curve for cells. This might be related to the fact that the mechanisms of formation of the cells and micrograins are different; micrograins are nuclei of primary recrystallization. To check this assumption, we compared the normalized statistical dependencies of dimensions of the micrograins in rolled nickel with the similar dependencies of dimensions of the recrystallized grains in rolled aluminum single crystal (Ref 9).

Table 1 Values of microstresses (σ, $\times 10^4$) and dimensions of regions of coherent scattering (L, μm) for nickel deformed by rolling and tension

Ni, ε%, kind of deformation	Tension, 11.2%	Tension, 23.7%	Fracture	Initial	Rolling, 50%	Rolling, 80%
σ	7.21	5.36	3.79	2.90	3.45	3.0
L	0.758	0.733	0.920	1.157	0.672	0.716

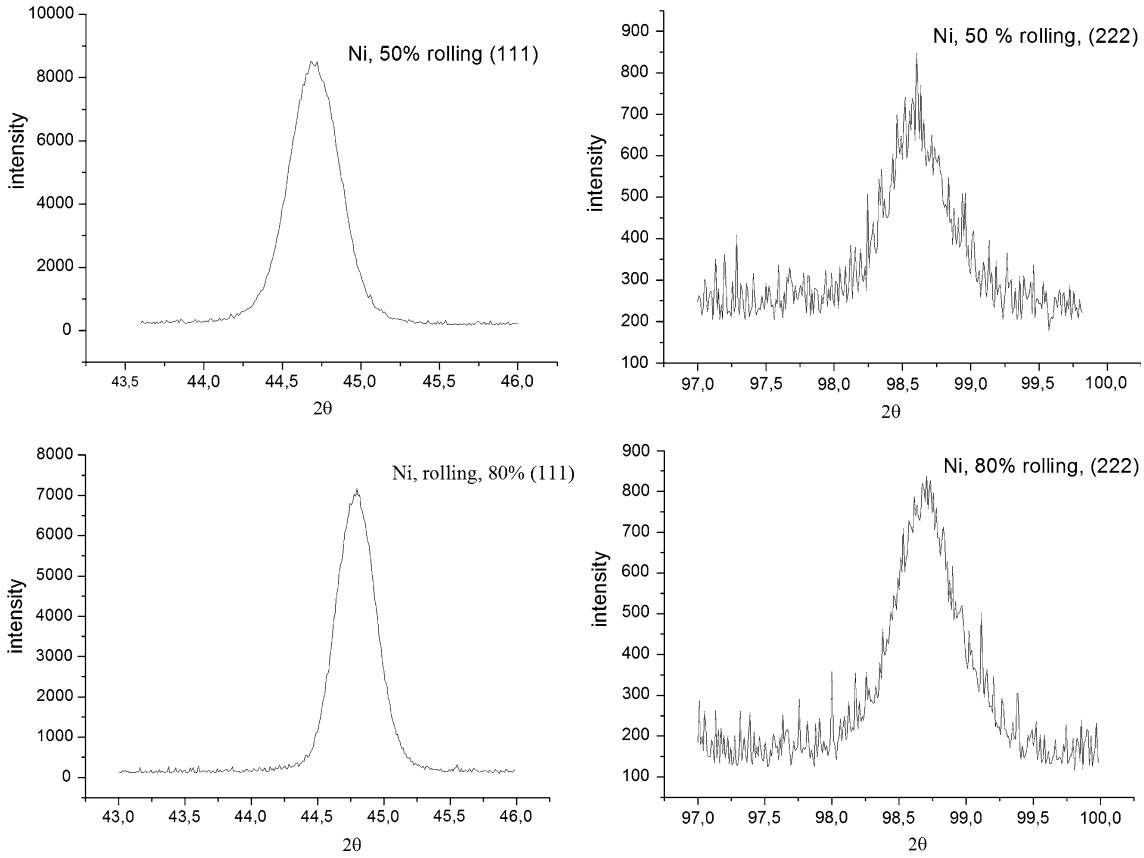

Fig. 1 Profiles of X-Ray peaks (111) and (222) for nickel deformed by rolling to 50 and 80%

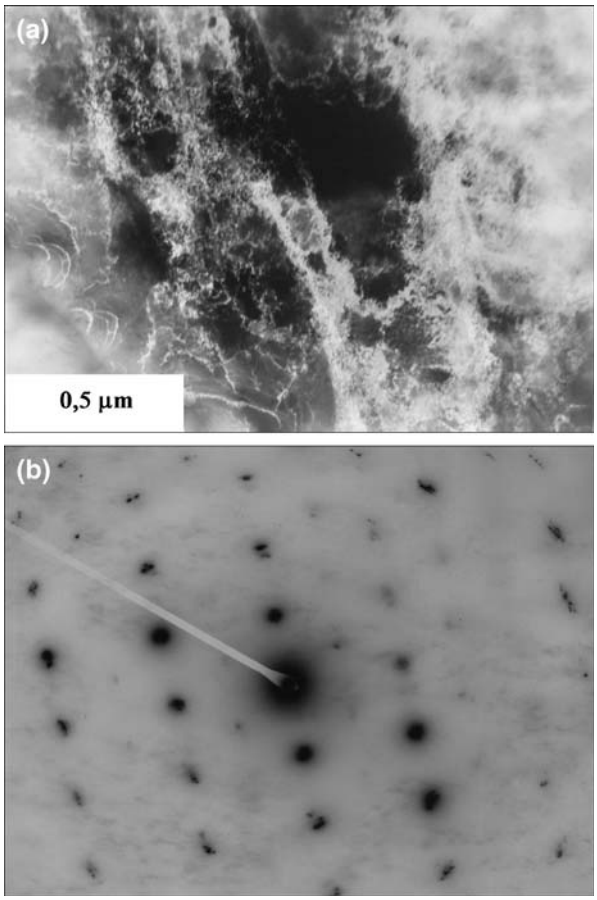

Fig. 2 Example of TEM structure of nickel after rolling to 50%; 1: cells, 2: micrograins, 3: microbands

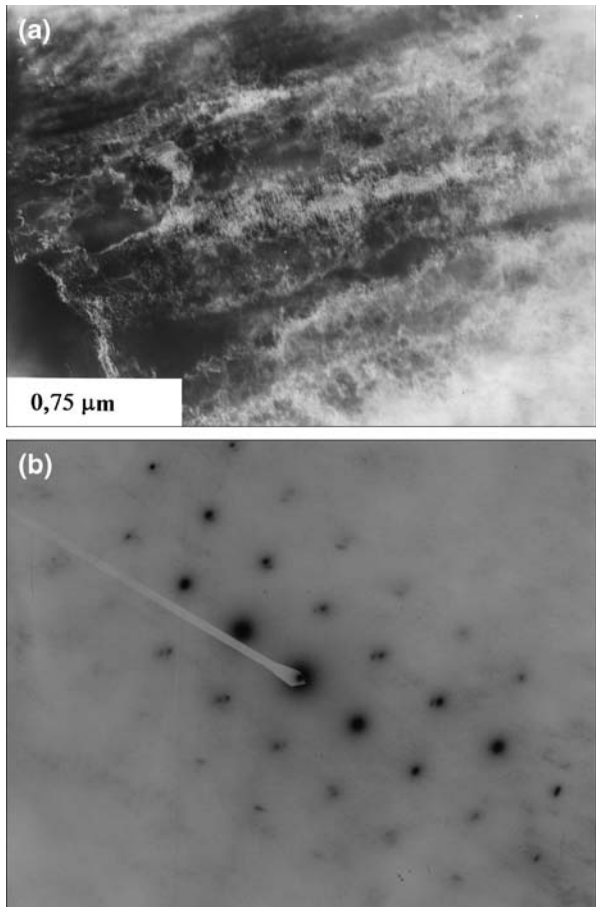

Fig. 3 Example of TEM structure of nickel after rolling to 80%; 1: cells, 2: micrograins, 3: microbands

In this study the quantitative investigation of the recrystallization kinetics on surface and in volume during rolling of aluminum single crystal {100} ⟨100⟩ at room temperature was performed. We found the qualitative appearances of dynamic and static recrystallization in rolling crystals during deformation process. The curves of recrystallized grains dimensions distribution in rolling and perpendicular directions were built. It was shown that the normalized curves of grains' dimensions distribution in all the cases may be described by only one function. The results were compared with well-known literary data and discussions from the point of view of contemporary representations about scaled invariance (scaling).

The resulting dependence of our data on dimensions of micrograins in nickel and recrystallized grains in aluminum single crystal is shown in Fig. 6.

It can be concluded from Fig. 6 that all the dimensions shown fall on one normalized distribution curve. This leads to a conclusion that micrograins are nuclei of primary recrystallization. This conclusion does not disagree with the above peculiarities of the micrograins: clearly defined boundaries and homogeneous internal contrast. The latter is attributable to a defect-free internal structure, which is characteristic of the recrystallization nuclei. And the clearly defined boundaries are indicative of the fact that micrograins (recrystallization nuclei) are separated from the single crystal matrix by the high-angle boundaries (see diffraction patterns on Fig. 2 and 3).

Therefore, our statistical analysis of dimensions of the cells and recrystallized grains in aluminum and nickel deformed by

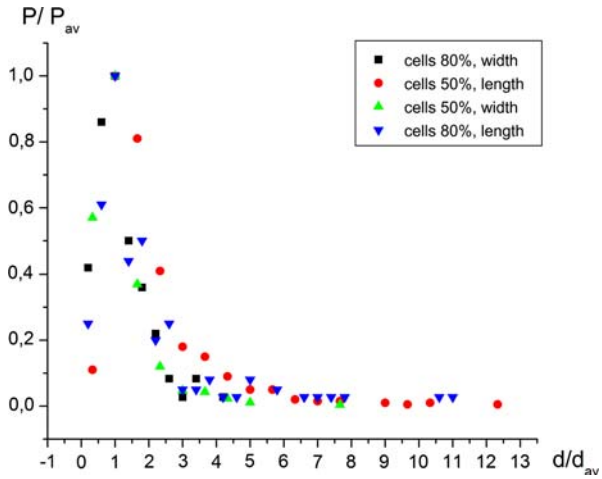

Fig. 4 Normalized curves of distribution in dimensions of the cells of nickel rolled to 50 and 80%: axis *X*—ratio of dimension of a cell in a selected direction to its average dimension; axis *Y*—repeatability (probability) normalized by the average value

rolling shows the presence of only one type of scale invariance of structural parameters. Quantitative similarity of the experimental curves of dimensional distribution of these parameters depends neither on the material studied (aluminum or nickel), nor upon the deformation degree (stage of the relaxation

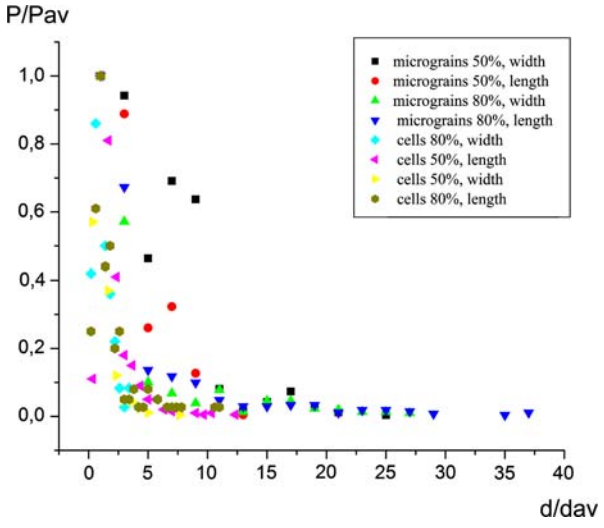

Fig. 5 Curves normalized by average dimension (axis X) and probability (axis Y) for distribution in dimensions of cells and micrograins of nickel after rolling to 50 and 80%

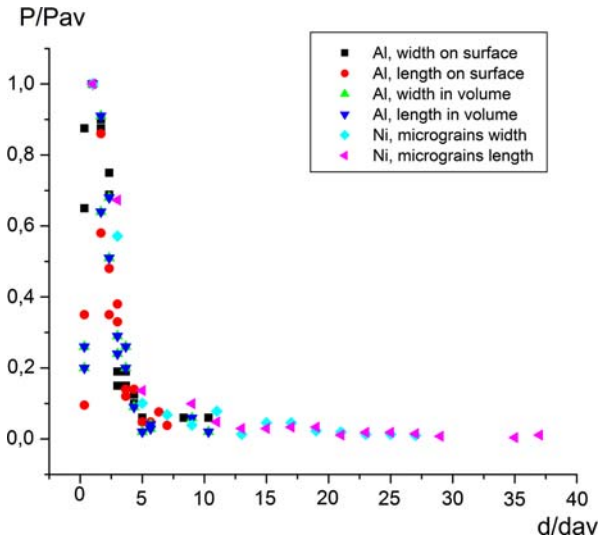

Fig. 6 Curves normalized by average size (axis X) and maximal repeatability (axis Y) for distribution of recrystallized grains in aluminum single crystal after deformation to 85% and micrograins in nickel rolled 80%

process). But the mechanism of relaxation process (recrystallization or cells formation) exerts its influence on the numerical characteristics of distribution curves (see Table 2).

It can be see that the numerical characteristics of normalized curves of distribution in dimensions of the cells in Ni to be different from similar characteristics of the curves for recrystallization grains in Al and micrograins in Ni.

The relaxation structures are found to be located primarily inside the microbands. In our opinion, it can be connected with that fact the microbands can be the synergetic structure elements which are formed during deformation. In the external stress field the material within the microbands is in a noncrystalline ("liquid-like") state. It assists in hydrodynamic localized plastic flow of material (see Ref 16, 17). After the load is removed, material inside the microbands may experience relaxation structural transitions ("crystallization") giving rise to many structural elements (mainly micrograins). So the formation of micrograins is the relaxation structure formation. The mechanism of its formation is similar one of primary recrystallization.

Table 2 Numerical characteristics of normalized curves of distribution in dimensions of the cells and recrystallized grains

	D_B	σ_B	m_3	m_4	as	e_k
Recrystallization (Al)	1.788	1.337	7.623	66.23	3.188	17.728
Recrystallization (Al) and micrograins (Ni)	1.96	1.40	8.55	75.9	3.12	16.76
Cells formation (Ni)	1.107	1.052	4.384	36.373	3.766	29.705

D_B dispersion, σ_B standard deviation, m_3 third moment, m_4 fourth moment, as asymmetry, e_k excess

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