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Effect of tension on the formation of local voids and integral damages in DC04 steel sheets

***A.Briukhanov*[1], *G.Gerstein*[2], *N.Volchok*[1], *F.Nurnberger*[2], *V.Lukashin*[1], *D.Dyachok*[1]**

[1]South Ukrainian National Pedagogical University, Odessa, Ukraine
[2]Institut fur Werkstoffkunde (Materials Science), Leibniz Universitat Hannover, An der Universitat 2, Garbsen, Germany

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The effect of tensile strains (ε) on changes of the damage level (D) for steel DC04 (0.06 % C, up to 0.35 % Mn, up to 0.40 % Si, ~ 0.025 % S and P) sheets was studied. D was evaluated by electron microscopy and the change in Young's modulus (E) of a conditionally intact sample (standard). Pre-recrystallization annealing results in the formation of a structure with a minimum D and increased E in the rolling direction (RD) and transverse direction (TD) with unchanged texture and anisotropy E ($E_{TD} > E_{RD}$). Tensile strain (TS) at the first stage (~ 5 %) leads to the formation of nucleation damage in the form of spherical pores up to 100 nm in size in the near-surface layer of steel, distributed over the volume and grain boundaries. E_{TD} and E_{RD} decrease to pre-annealing values. At a deformation of 10–20 %, new pores and chains of them are formed. E decreases and D increases uniformly. At deformations of more than 20 %, the pores in the chains show a tendency to merge (coagulate) with the formation of cracks in the form of plates, which are perpendicular to the direction of (TD). The decrease in the Young's modulus in RD and TD and the increase in D are sharper. An analysis of texture changes confirms the predominant contribution of damage to the effect of a decrease in Young's modulus under tensile deformation.
Keywords: deformation, tension, damage, Young's modulus, texture, pore, microcrack.

Вплив розтягування на утворення зародкових пошкоджень і інтегральна пошкодженість листів сталі DC04. *А.Брюханов, Г.Герштейн, Н.Волчок, Ф.Нюренбергер, В.Лукашин, Д.Дячок*

Вивчено вплив деформацій розтягуванням (ε) на зміни рівня пошкодження (D) сталі DC04 (0.06 %, до 0.35 % Mn, до 0.40 % Si, ~ 0.025 % S і P). D оцінювали методами електронної мікроскопії та зміни модуля Юнга (E) відносно умовно непошкодженого еталону. Дорекристалізаційний відпал формує структуру з мінімальним D і підвищеними E в напрямку прокатки (НП) та поперечному напрямку (ПН) при незмінній текстурі та анізотропії E ($E_{ПН} > E_{НП}$). Деформація розтягуванням (ДР) на першому етапі (~ 5 %) призводить до утворення в приповерхневому шарі сталі зародкових пошкоджень у вигляді пір, розмірами до 100 нм сферичної форми, розподілених за обсягом та границями зерен. $E_{ПН}$ і $E_{НП}$ зменшуються до значень, що були до рекристалізації. При деформації 10–20 % утворюються нові пори та ланцюжки з них. E зменшується, а D збільшується рівномірно. При деформаціях більше 20 % пори в ланцюжках виявляють тенденцію до злиття (коагуляції) з утворенням тріщин у вигляді пластин, які перпендикулярні напрямку (ДР). Зменшення модуля Юнга в НП та ПН та збільшення D відбуваються різкіше. Аналіз текстурних змін підтверджує домінуючий внесок пошкоджень до ефекту зменшення модуля Юнга при деформації розтягуванням.

1. Introduction

Microdamages in the form of microvoids, open microcracks, shear bands are almost always present in engineering materials even before the application of an external load [1]. In the area of viscoelastic deformation, they become provocateurs of the emergence and development of fracture centers. Therefore, the study of the conditions for the occurrence of microdamages, depending on the initial structure, type and degree of subsequent loading, opens up opportunities to develop calculation methods for controlling the porosity of structural materials and, accordingly, their service life [2]. The study of the dynamics of the nucleation, growth, and coagulation of microdamages in metal polycrystals at different stages of plastic deformation makes it possible to evaluate their service life [2], as well as to develop strength criteria (SC) taking into account damage structure (porosity) at the mesolevel. Prediction of the fracture process in brittle materials is based on the results of calculating the critical energy density at the particular crack tip [3]. However, the effectiveness of this approach decreases in the case when the fracture process is accompanied by simultaneous plastic deformation. Intense nucleation and coagulation of various kinds of discontinuities occurs inside grains, at the grain boundaries of a polycrystal, on impurities, etc. A field of internal stresses is formed and local cracks appear, the growth of which leads to the destruction of the solid.

For a quantitative assessment of the integral damage of a polycrystalline, in the continuum mechanics of damage, based on the works of Kachanov L.M. [4] and Rabotnova Yu.N. [5], a variable parameter is introduced $D = \delta \tilde{S}/\delta S$, where $\delta \tilde{S}$ is the effective area of the intersections of all microcracks and microcavities, which lie in the plane of the intersection with a Representative Volume Element (RVE) at the point M of a damaged body; δS is the area of this intersection.

The coefficient of damage D, may be found from the electron-microscopic studies of the structure of RVE in a polycrystalline solid [6]. This approach requires a large amount of experimental research, careful preparation of samples and highly qualified research personnel. In addition, there is no criterion for the representativeness of RVE has been established for describing damage by methods of statistical averaging of local damage concentrations [7]. Thus, the question of transferring the results obtained by electron microscopy methods from different samples (differential damage) to the entire volume of the material remains open. This question is fundamental in the constitutive theories of strength with allowance for damage.

With a relatively uniform distribution of damage over the volume of the material, rather effective methods for assessment of the level of integral damage are indirect methods based on measuring properties that reflect changes in the structure of the polycrystal under study [6]. For viscoelastic materials, such properties are elastic properties. The ratio of the modulus of normal elasticity of a material in a damaged state $\tilde{E}$ to the modulus of an undamaged specimen (reference). $\tilde{E}$characterizes the level of material damage in relation to this reference:

$$D = 1 - \frac{\tilde{E}}{E}. \quad (1)$$

Determination of the elastic moduli is possible by non-destructive methods based on the measuring the frequency of natural vibrations of the samples or on the results of changes in the propagation speed of ultrasonic (US) waves [8, 9]. Microdamages due to plastic deformation of metallic materials, for example, when straightening rolled steel and further stamping products from it, significantly change the level of strength and plasticity properties of the material. Therefore, it is important to take into account this type of defective structure of deformed materials in order to predict structural durability.

The purpose of this work is to study, using electron microscopy, the nucleation and development of local damage in sheets of steel DS04 after tensile deformation, as well as to evaluate the integral damage by changing the elastic moduli.

2. Materials and experimental technique

The material of the research was steel sheets DC04 (0.06 % C, for 0.35 % Mn, for 0.40 % Si, ~ 0.025 % S and P) from Salzgitter Flachstahl, 1.5 mm thick with a single-phase ferrite structure [10]. The company guarantees the declared chemical composition, yield strength ($\sigma_{0.2}$) ~ 210 MPa, tensile strength (σ_b) within 270–350 MPa, elongation at fracture > 38 %, density — 7870 kg/m^3.

Control tensile tests of 3 samples with a working part length of 50 mm, a width of 20 mm were carried out at a tensile speed

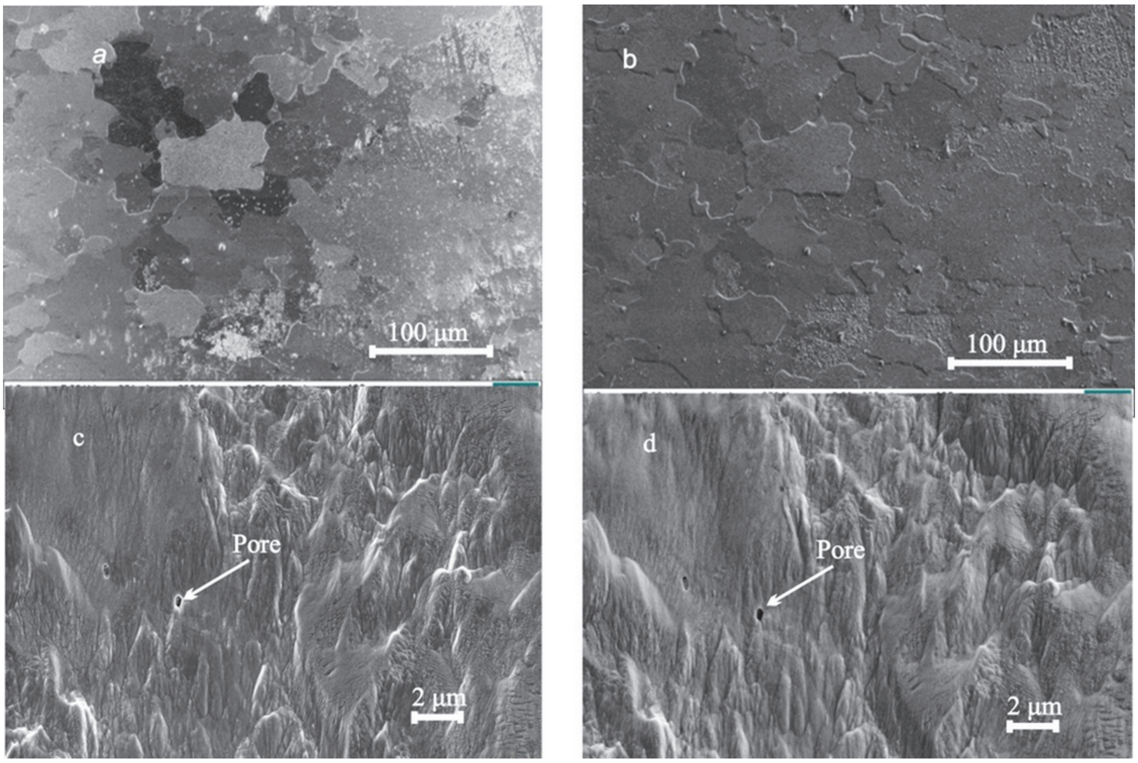

Fig. 1. SEM image of the structure in the perspective — (a, c) "inlense" and in (b, d) — side view of the DC04 steel sheets after pre-recrystallization annealing; (a, b) 200, (c, d) 5000.

of 0.5 mm/min (Fig. 1) and showed the following: $\sigma_{0.2}$ = 213.8; σ_b = 307.1 MPa, relative uniform elongation (A_g) — 22.4 %; relative elongation at fracture — 46.7 %. The uniform elongation is broken after deformations of ~ 22–25 %.

Initial steel sheets in the form of plates, 200×200 mm in size, were annealed at a temperature of 350°C for 12 hours in a neutral gas (argon) to obtain a structure with a minimum level of damage [11]. Then, 5 groups of samples were formed, each of which contained samples of the two main directions of the sheet. Samples of the first group were subjected to tension deformation up to 5 %, the second — up to 10 %, the rest — up to 15, 20, and 25 %. Separately, some samples were deformed up to 35 % for EM studies of the structure.

Samples in the form of "dogs bone" were cut from the plates in the rolling direction (RD) and the transverse direction (TD); then they were subjected to various degrees of tensile deformation at a speed of 0.8 mm/min on a Zwick Z100 universal testing machine (100 kN). The microstructure and texture of the steel samples in the initial state and after tensile deformations were studied by scanning electron microscopy using an FE-REM device (ZEISS Supra 55 VP).

After mechanical polishing with lubricant up to 1 μm and additional polishing in the vibration polishing unit Vibro Met 2 (Buehler Co), the samples were subjected to a special ionic cutting combined with intense ion polishing in a neutral gas flow (cutting at an angle with an ion beam allows precise placement of microcuts in near-surface regions). The ion flow was directed by a tungsten wedge at an angle of 30° to the sample plane.

The secondary electron detector and the Inlens detector were used to scan the microstructure: the first one was used to check the reliability of information about the presence of a pore, and the second one was used to obtain a high-contrast picture. As a representative area on the surface of the sample, a square of size XHY was selected, which was divided into cells of size 5×5. Electron microscopy images were obtained from each of the cells.

The annealed steel has a ferritic structure typical of low-carbon steels with an average grain size of ~ 80 μm (Fig. 1a, b) with a minimum level of damage.

There are some discontinuities in the form of rare pores, which apparently remained after industrial processing of sheets

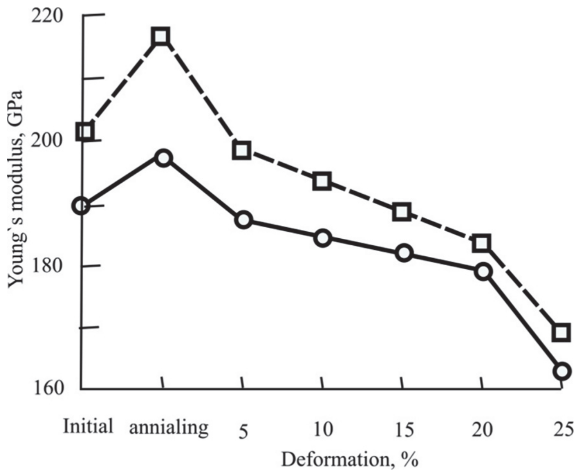

Fig. 2. Changes in Young's modulus of DC04 steel sheets under tensile deformation: ○ — in RD, □ — in TD.

(Fig. 1c, d) and which are seen only at high magnifications (>×5000).

Young's modulus was determined from the frequency of natural transverse vibrations of flat samples. They were cut from the middle part of the "dog bones". Rectangular samples were processed in a bag to ensure the equal dimensions. The natural vibrations of the sound frequency were excited in a sample with a density ρ, length l, thickness d. The natural frequency was measured by the resonance method [12] using the Spectra PLUS computer program [13]. Young's modulus was calculated by the formula [8]:

$$E = \frac{48\pi^2}{m^4} \cdot \rho\frac{l^4}{d^2}\nu^2, \qquad (2)$$

where m is the Modelung constant equal to 4.72 for the main tone.

The study of the influence of deformation on the change in the elasticity modulus is reduced to measuring the change in the frequencies of natural vibrations on the same sample. Only the change in thickness after stretching is taken into account. Modern computer methods make it possible to record a change in frequency in the range of 500–1000 Hz with an accuracy of 0.01 Hz.

3. Results and discussion

Fig. 2 shows the curves of changes in Young's moduli in RD and TD after annealing of steel sheets and subsequent tensile deformations. Young's modulus of the original steel sheets in the TD is higher than in the RD. This is typical for bcc iron polycrystals with a recrystallization texture [14]. The pre-crystallization annealing of steel sheets leads to an increase in Young's modulus in both directions of the sheets (~ 4 and 7 % in RD and TD, respectively). After 5 % stretching, Young's modulus decreases to values somewhat lower than in the original sheets.

After deformation up to 10, 15, and 20 %, Young's moduli decrease more or less monotonically by about 5 and 12 % in RD and TD, respectively, relative to a deformation of 5 %. Further, the monotonicity of the change in E is broken, and the decrease in Young's modulus after deformation to 25 % with respect to the previous tension (20 %) is 5 and 6 % in RD and TD, respectively. Further deformation leads to the formation of a neck, and the measurement of Young's modulus by the method of natural vibrations of flat samples becomes ineffective.

Tensile deformations change the EM pattern of steel structures (Fig. 3,4). Deformation up to 5 % leads to the formation of pores distributed over the surface of the grains. The pore shape is close to spherical, the sizes do not differ significantly from each other.

Deformation up to 10 % leads to the formation of new spherical pores. At deformation degrees of 15 and 20 %, chains of pores are formed in the regions near the grain boundary, which are close to each other, but do not yet merge into cracks. The pores scattered over the surface of the grains do not significantly change their shape and size. New pores appear, thereby increasing the integral density of damage. The chains of pores, which do not yet show signs of cracks, form clusters in the form of plates oriented perpendicular to the direction of tension (DT).

At deformations of ~ 20 %, there is a tendency for individual closely spaced pores to merge (coagulation). At higher strains (~ 30–35 %), the uniformity of deformation is disturbed, a "neck" appears on the samples, and damages on the surface of the samples in the form of roughness become visible.

The sizes of damages distributed over the surfaces of grains, can be estimated by high magnification SEM images (Fig. 4) as 100–200 nm. According to the classification [15], they belong to germinal pores, are localized in the near-surface layer, and have a spherical shape. According to [16], the formation of such micro-discontinuities is possible due to dislocation or vacancy blunting

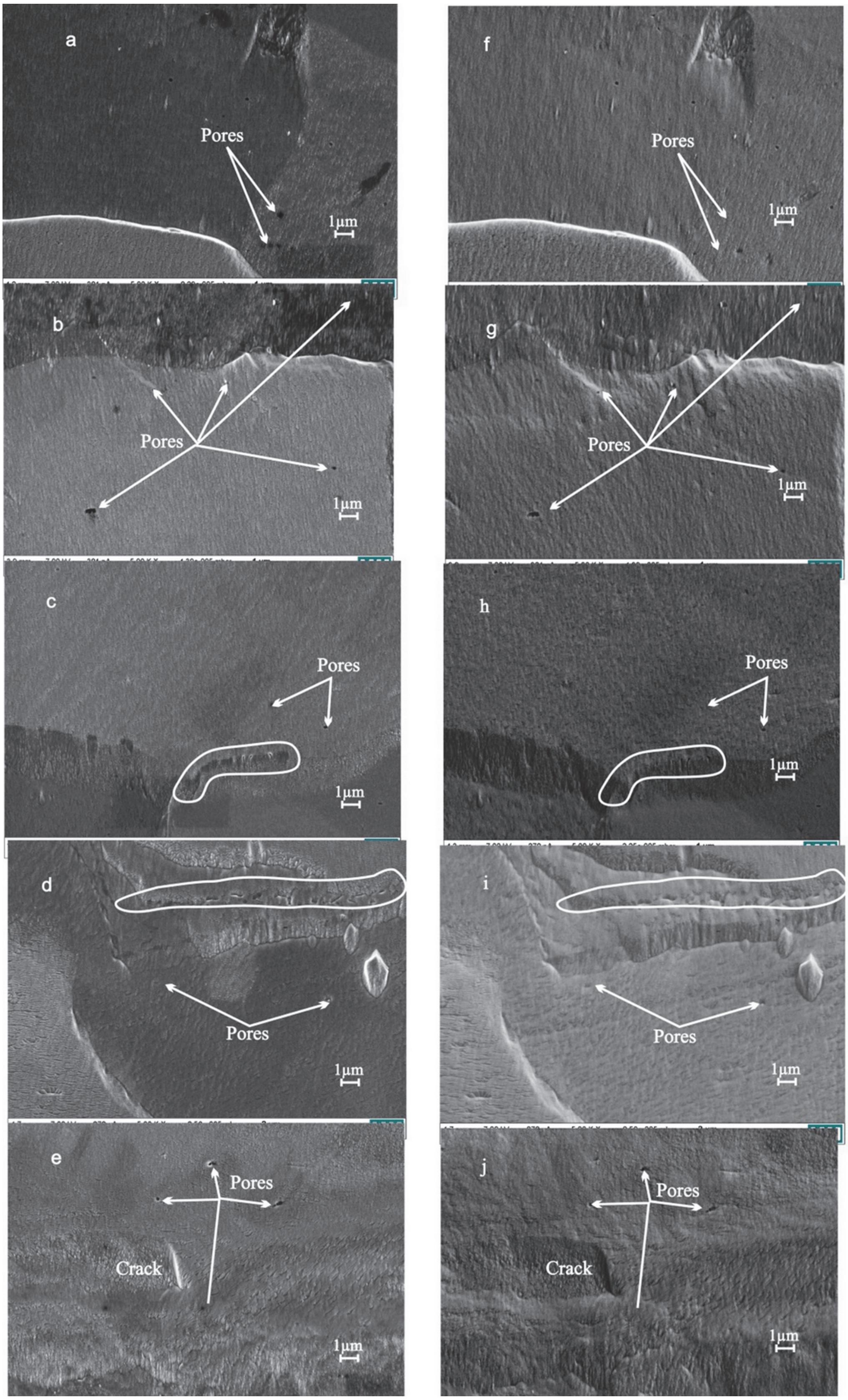

Fig. 3. SEM image of the structure in foreshortening — (a, b, c, d, e) "inlense" and in (f, g, h, i, j) — side view of DC04 steel sheets after pre-recrystallization annealing and tensile deformation to a, f — 5 ; b, g — 10; c, h — 15; d, i — 20; e, j — 35 %.

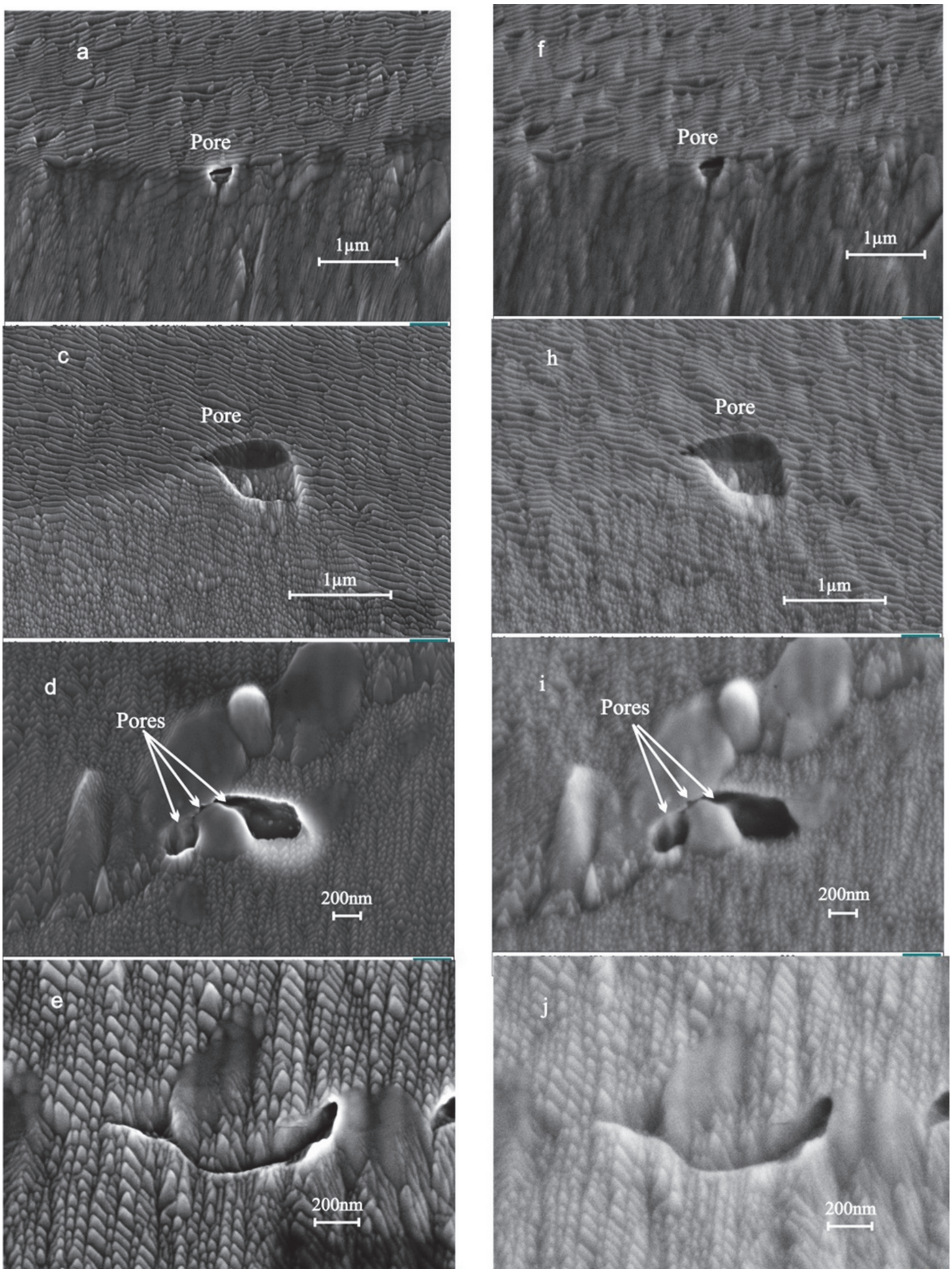

Fig. 4. SEM image of the structure in foreshortening — (a, c, d, e) "inlense" and in (f, h, i, j) — side view of DC04 steel sheets after pre-recrystallization annealing and tensile deformation to a, f — 5; c, h — 15; d, i — 20; e, j — 35 %.

of wedge-shaped microcracks as a result of the collapse of opposite dislocation clusters into a crack. Another case (dislocation model) [17] assumes an "explosive" nucleation of germinal discontinuities as a result of fluctuations causing the coalescence of head dislocations. The initiation of damage observed in DC04 steel under tensile deformation fits into this model. Voids, close to spherical in shape, voids on the grain surface is observed after stretching by 5 %.

An increase in the degree of deformation does not lead to a significant increase in the pore size. New small damages are formed, scattered over the surface of the grains and at their boundaries. We did not observe a significant number of wedge-shaped microcracks. In the range of these deformations, Young's modulus decreases monotonically, without jumps. Variable *D*, calculated according to (1) (Fig. 5), monotonically increases, but in the area of deformations of more than 20 %, the damage increases rapidly.

With these deformations, there is a tendency for closely spaced pores to coalesce (coagulate). This is clearly seen in Fig. 3d, i, where two large pores merge due to the destruction of the bridges between the three small pores ~ 100 nm in size located between them. Tensile deformation up to 35 % leads to the formation of clear cracks against the background of a more or less uniform distribution of pores along the plane of the grains. The images with magnifications of 20000–55000 show that tensile deformations distort the shape of the pores, however, the latter still show a tendency to maintain equiaxiality (Fig. 1a, f, c, h). These figures clearly show the rough surface of the grains after ion polishing.

It is interesting, that the surface of the lower side of a pore is also rough, like the surface of a grain, i.e. this means that this is the surface of the next fragment or block. This testifies in favor of the fact that after large active deformations within the boundaries of misoriented fragments, the dimensions of which are commensurate with the dimensions of the blocks, voids with a size of about 0.1 μm appear [18]. The crack formed at deformations of ~ 35 %, has the shape of a curved plate oriented perpendicular to (DT) (Fig. 2e, j). The shape and orientation of the crack corresponds to the Gurson-Tvergaard-Needleman (GTN) model for elastically viscous materials [19, 20].

In the range of deformations, at which the coalescence of chains of equiaxed pores into cracks occurs, there is a sharper decrease in Young's modulus (Fig. 2); correspondingly, the damage constant *D* increases to values of 14, and 24 % in RD and TD, respectively (Fig. 5). The changes in Young's modulus reflect the process of damage accumulation during tensile deformation.

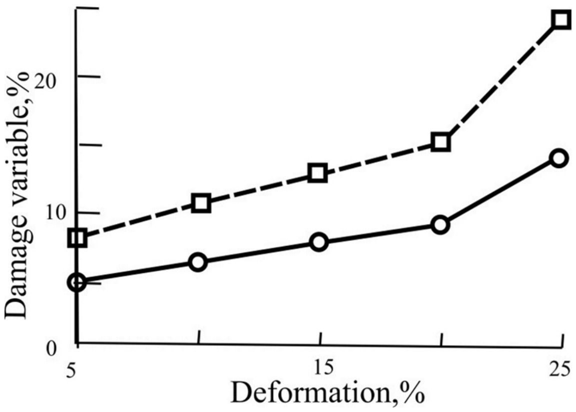

Fig. 5. Change of the integral damage variable of DC04 steel sheets due to tensile deformation.

If at each stage of deformation there is a total deformation expressed as a sum of elastic (ε^e) and plastic (ε^p) components [6], then Young's modulus, measured by the resonance method according to the frequency of natural transverse vibrations of the plates, characterizes the elastic part of the deformation for each of the states:

$$\varepsilon^e = \frac{\sigma}{E_D}, \tag{3}$$

σ is the stress required to achieve a specific uniform deformation and is the Young's modulus of the deformed plate, the changes in which under the corresponding strains and stresses characterize the areas of formation, accumulation of damage and their transformation into accumulations and microcracks. The texture factor also has a significant effect on the change in the elastic moduli [14].

The effect of tensile deformation on the texture of DC04 steel sheets was studied in [21]. The original and annealed DC04 steel sheets had a recrystallization texture with a high degree of scattering. The texture is multicomponent. The predominant ideal orientations (IO) in the plane of the sheet are isotropic orientations $\{111\}\langle \underline{U}VW\rangle$ and close to $\{554\}\langle 225\rangle$ and $\{108\underline{7}\}\langle 135\rangle$. Orientations $\{335\}\langle 7123\rangle$ and $112\langle 681\rangle$, as well as orientations $\{100\}\langle 001\rangle$ and $\{100\}\langle 012\rangle$ are present as minor ones.

Changes in the texture of DC04 steel sheets after tensile deformation are characterized by the fact that slip orientations are superimposed on the set of orientations of the texture of recrystallization and rolling present both in the original and annealed sheets

[21], which change the nature of the anisotropy of the elastic properties of the sheets.

The main orientations determining the anisotropy are sharply anisotropic orientations {110} <*UVW*>. These orientations have a large average value of the modulus in the plane of sheets for orientations of the {100} <*UVW*> type and approximately the same as {111} <*UVW*> [14]. However, the orientations of the {111} <*UVW*> type in the texture did not increase after stretching. Therefore, the contribution of damage to the decrease in Young's modulus dominates. Further, the formation of cracks creates zones of probable redistribution of stresses, leading to their growth and destruction in accordance with the existing classical continuum theories [3–6], and the appearance of pore chains seems to be a precursor of classical fracture.

4. Conclusion

1. Sheets of DC04 steel (0.06 % C, up to 0.35 % Mn, up to 0.40 % Si, ~ 0.025 % S and P) after annealing at 350°C are characterized by a low level of damage, texture with a predominance of isotropic ideal orientations (IO) of the {111}<*UVU*> type, secondary {335}⟨7123⟩, 112⟨681⟩ and {100}⟨001⟩, {100}⟨012⟩, high Young's modulus of 198.13 and 216.3 GPa in the rolling direction (RD) and transverse direction (TD), respectively.

2. Tensile deformation up to 5 % leads to the formation of germinal damages in the near-surface layer of steel in the form of pores, up to 100 nm in size, close to a spherical shape, distributed over the surface and grain boundaries. Young's moduli in RD and TD decrease to pre-combustion values.

3. Deformation up to 10 % leads to the formation of new germinal discontinuities in the metal. At degrees of deformation (15 and 20 %), chains of pores are formed in the areas near the boundary between the grains without cracks. New spherical pores of approximately the same size appear. Young's moduli decrease monotonically, and the integral damage also increases uniformly.

4. At deformations of more than 20 %, there is a tendency for individual pores in the chains to coalesce (coagulation) with the formation of cracks in the form of plates, perpendicular to the direction of tension (DT). In this deformation range, the decrease in Young's modulus in RD and TD and the increase in damage occur more sharply.

5. Changes in the texture of DC04 steel sheets after tensile deformation are characterized by the development of sharply anisotropic orientations {110} <uvw>, the combined effect of which with the IO does not decrease the value of E. The contribution of damage to the decrease E_s is the Young's modulus is predominant, and the moment of merging of pore chains into cracks a sign of the fracture process.

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