

# MICROSTRESSES DEVELOPING UNDER SEVERE COLD DRAWING IN NANOSCALE PEARLITIC STEEL

N. Yu. Zolotarevsky<sup>1</sup>, Yu. F. Titovets<sup>2</sup> and D.M.Vasiliev<sup>1</sup>

<sup>1</sup>Physical-Mechanical Department of St. Petersburg State Polytechnical University, Politechnicheskaya 29, St. Petersburg, Russia

<sup>2</sup>Institute of Problems of Mechanical Engineering, Russian Academy of Sciences, Bolshoj 61, Vasil. Ostrov, St. Petersburg 199178, Russia

Received: July 27, 2004

**Abstract.** The influence of cold drawing on the internal stresses (microstresses) developed in the ferrite phase of a pearlitic steel due to the plastic interaction of ferrite and cementite has been studied using X-ray technique. The microstresses were evaluated from experimental data by the method accounting for internal stresses caused by the ferrite plastic anisotropy (referred to as mesostresses). The main result is that microstresses in the ferrite phase gradually decrease down to near-zero level with increasing plastic strain.

## 1. INTRODUCTION.

Heavily drawn pearlite represents, as it was noted in [1], a nano-composite with thickness of ferrite and cementite lamellae decreasing with drawing. The strength of the drawn pearlite increases up to very high level with this lamellar structure evolution [2]. At the same time, significant ductility is retained. A remarkable peculiarity of the drawn pearlite is that the reduction of sample cross area under tensile testing, a parameter characterizing local ductility, increases at the true strain below from 1.5 to 2 [1].

The increase in local ductility is known to be associated with the decrease of internal stress concentration [3]. The question is: the stress of what nature plays key role herewith? Recently the local ductility increase was presumably related to the metallographic texture development [1], that is to the fact that stress concentration appearing due to incompatibility of colonies deformation decreases when lamellae become oriented along the wire axis. Another explanation [2] notices a significant change

of the cementite structure in the course of drawing. This change is expected to result in the decrease of interphase stress concentration appearing due to plastic interaction of the hard phase (cementite) and the soft phase (ferrite). Naturally, both these possibilities can contribute to the local ductility increase.

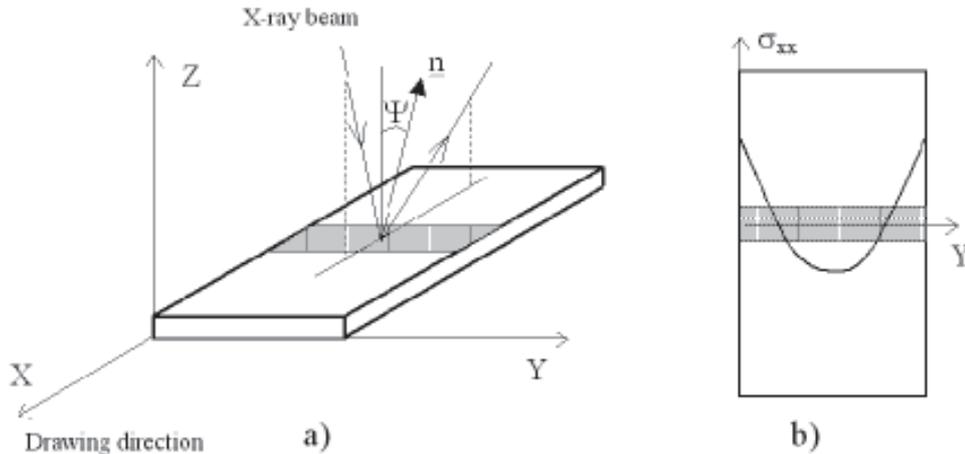
Present study of the interphase stresses in the heavily drawn pearlite is aimed at clarification of mechanisms controlling its plastic behavior and unique mechanical properties. The stress evolution with increasing true plastic strain from 0.25 to 2 will be analyzed and discussed.

## 2. EXPERIMENTAL

The material studied was commercial steel containing approximately 0.8% of carbon. The initial rods (before cold drawing) were obtained by hot rolling to diameter 11.18 mm. Then, the rods were cold drawn in 8 steps to final wire diameter 4.13 mm (true strain  $\epsilon$  of 2). Some details concerning the drawing process and the texture and residual stresses distribution over the wire cross section were described ear-

---

Corresponding author: Yu. F. Titovets, e-mail: titovets@ phmf.spbstu.ru



**Fig. 1.** Design of the X-ray experiment. The region exposed to the X-ray beam is shadowed. (a) – view of sample; (b) – distribution of axial macrostress.

lier [4]. It was shown that significant texture and stresses non-uniformity exists only in the narrow surface layer of about 0.1 mm width. This layer was removed during preparation of specimens for X-ray study.

The X-ray experiment has been designed to provide us information only about microscopic stresses in the phase analysed. Plate samples have been obtained from the wires with various strain levels (Fig. 1a). The samples have been prepared by grinding on diamond abrasive disk with water cooling and subsequent etching to remove work-hardened layer. The plate width is equal to the wire diameter, while the thickness is at least three times smaller. In such a plate the macrostress distribution can be considered with sufficient accuracy as dependent on  $Y$  coordinate only. Under condition, that X-ray beam overlaps the plate totally in the transverse direction (Fig. 1b), the axial macrostresses  $\sigma_{xx}$  are balanced within the irradiated volume, that is the mean macrostress over this volume is equal to zero.

The interplanar spacing  $d$  for planes  $\{220\}$  and  $\{211\}$  was measured in various directions characterized by the azimuth angle  $\varphi$  and the angle  $\psi$  between reflecting planes and the sample surface (Fig. 1). The measurements were performed for  $j=0$  corresponding to the drawing direction. Diffraction angles  $2\theta$  were measured both for  $+\psi$  and  $-\psi$  rotations; mean values were then taken for analysis as usually [5]. Peak positions were determined by mean of position-sensitive detector using  $K\alpha\text{Fe}$  radiation for  $\{220\}$  reflections and  $K\alpha\text{Cr}$  for  $\{211\}$  reflections. The variations of interplanar spacing were obtained from the measured diffraction angles  $2\theta$  by help of the relationship  $\theta = (d-d_0)/d_0 = -\text{ctg}\theta$  ( $\theta-\theta_0$ ),

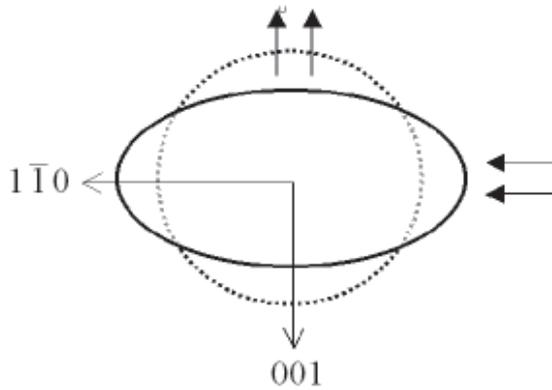
where  $\theta_0$  and  $d_0$  correspond to the stress-free crystal. The X-ray measurements were carried out on the wires drawn with eight steps of around 0.25 up to the true strain of 2.

Additionally, the measurements were conducted on the annealed wire. Samples of heavily drawn wire ( $\epsilon = 2$ ) were annealed during 5 h. at 400 °C to remove residual stresses developed under drawing. Then, these samples were deformed by tension to various strains, and, again, residual stresses were examined.

## 2.1. Method of microstresses evaluation

In the present study, microstresses of two kinds affect X-ray diffraction. The first are the interphase stresses partitioned between the ferrite and cementite phases. The second are the stresses appearing due to plastic interaction of ferrite crystallites of different lattice orientation [5]. It was shown [5-7] that last ones cause a strong non-linearity of  $\epsilon\text{-sin}^2\psi$  curves in the case of heavily drawn pearlite. Since these orientation dependent stresses appear on the scale of ferrite mesostructure formed within separate pearlitic colonies [8], we called them the mesostresses. A method was suggested [6,7] allowing to determine the stress state in the case of steel wire drawing, when the  $\langle 110 \rangle$  fiber texture is developed.

It is known [9] that a non-constrained ferrite crystal deformed by tension in the  $[110]$  direction would demonstrate plain deformation, namely, thinning in the  $[001]$  direction with retaining size in the  $[1\bar{1}0]$  direction, see Fig. 2. Under constraint, a reactive



**Fig. 2.** Cross section of the crystallite having the  $[1\bar{1}0]$  direction parallel to the wire axis. The compressive stress along  $[1\bar{1}0]$  and the tensile stress along  $[001]$  (arrows indicate stress direction) appears in the crystallite due to reaction of its neighborhood to its non-axisymmetric deformation.

stress appears aimed to provide axisymmetric deformation of the crystallite. It consists of a tension along the  $[001]$  direction and compression along the  $[1\bar{1}0]$  direction. It was assumed that these two stress components are equal in absolute value, signed  $\sigma_{or}$ . The procedure of residual stresses evaluation consists in four main steps:

1. Measurement of a  $\varepsilon\text{-sin}^2\psi$  curve at  $\varphi=0$  for any reflection.
2. Measurement of quantitative characteristics of axial texture – a degree of acuteness components  $\langle 110 \rangle$ .
3. Modeling this curve taking into account the axial interphase stress, and also the orientation dependent stress tensor defined above in the crystal basis. Reuss approach was used regarding the elastic anisotropy of ferrite lattice and the orientation distribution (crystallographic texture) [7].
4. Determination of the two unknown stress values from the best fit of the calculated curve to the experimental data.

The orientation distribution for the  $\langle 110 \rangle$  fiber texture is then to be known for the stress evaluation. This distribution is assumed to meet the normal distribution for the angle of deviation of the  $\langle 110 \rangle$  direction from the wire axis. Its spreading (characterized by its standard deviation) is the only parameter to be known. It was shown [10] that for the

steel wires examined the spreading varies from about  $20^\circ$  for  $e=0.25$  to  $7^\circ$  for  $e=2$ .

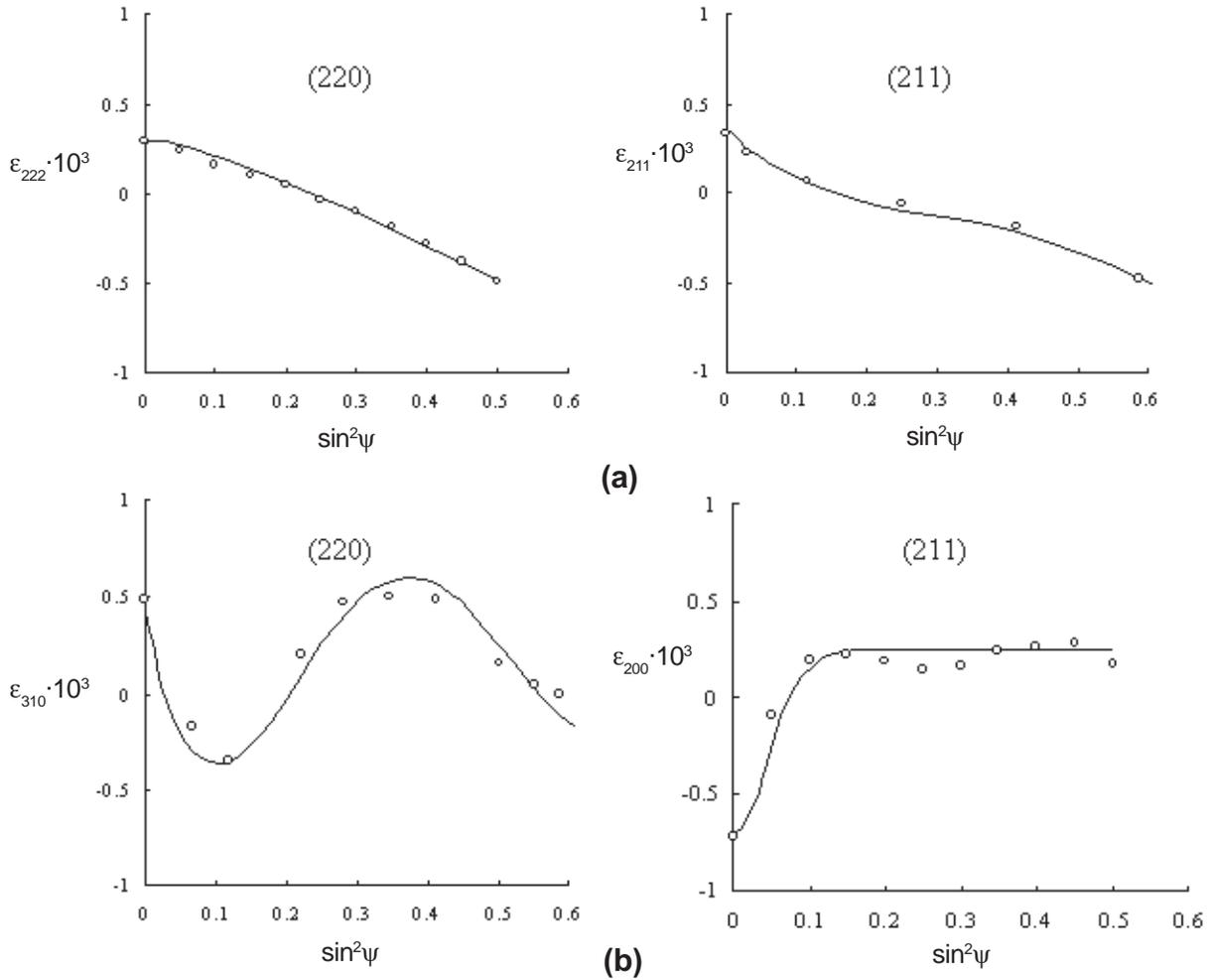
In the present simplified approach, the orientation dependent stress tensor is considered to be constant in the crystal basis. This assumption is reasonable in the case of a sharp  $\langle 110 \rangle$  texture, when the contribution of crystallites highly deviated from this ideal orientation is not significant. Hence, only in this case the model provides adequate estimation of  $\sigma_{or}$ , while for small drawing strains the estimation is to be considered rather as a model parameter than actual stress level. At the same time, the model enables to describe non-linearity of the  $\varepsilon\text{-sin}^2\psi$  dependencies for the whole range of drawing strains, and by that way to determine the interphase stress ( $\sigma_{ph}$ ), that is the aim of the present work.

Examples of  $\varepsilon\text{-sin}^2\psi$  curves are shown in Fig.3, where the curves for minimal drawing strain  $e=0.25$ , Fig. 3a and drawing maximal strain  $e=2$ , Fig.3b are represented. Theoretical fit is shown there together with experimental points. After small drawing strain, a slope of the curves indicates considerable compressive interphase stress in ferrite, while non-linearity peculiar to a corresponding reflection is negligible. Alternatively, after large drawing strain strong non-linearity appears due to development of a pronounced crystallographic texture. In the latter case  $\sigma_{or} = 100$  MPa for  $\{220\}$  reflection and 120 MPa for  $\{211\}$  reflection.

### 3. RESULTS

Variation of the interphase stress determined according the method described in the previous section is presented in Fig. 4. One can see a significant reduction of the stress with drawing strain. While its average (for  $\{220\}$  and  $\{211\}$  reflections analysis) value is 285 MPa for  $e=0.25$ , it decreases down to 25 MPa for  $e=2$ . Herewith the decrease approaches its saturation about after  $e=1.5$ . As it is seen from Fig.4, the behavior of interphase stress correlates with variation of the reduction of cross area.

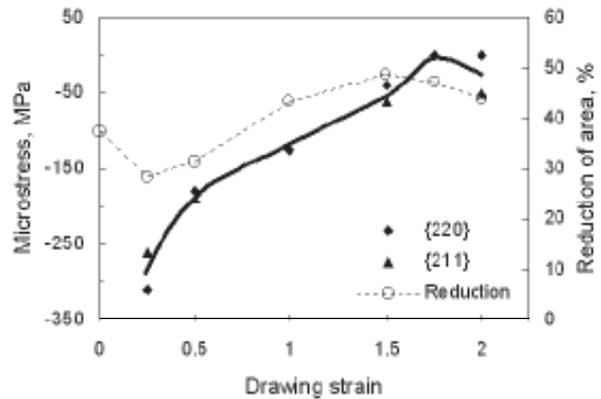
It is worth noting that the level of compressive interphase stress at  $e=0.25$  is comparable with the saturation level achieved by tensile deformation of lamellar pearlite, about 350 MPa, observed by Belassel *et al.* [11]. The pearlitic steel samples studied by Belassel *et al.* had no metallographic neither crystallographic texture. The question arises: how the pronounce texture developed in the heavily drawn wire will influence the stress behavior at small strains?



**Fig. 3.** Measured lattice strains  $\epsilon$  vs.  $\sin^2\psi$  (circles) obtained for {220} and {211} reflections, and theoretical fit (full line) to the experimental data. (a) -  $\epsilon=0.25$ ; (b) -  $\epsilon=2$ .

In order to answer the question, additional experiment has been carried out. Samples of heavily drawn wire ( $\epsilon = 2$ ) were annealed during 5 h. at 400 °C. It was shown earlier [12,13] that the annealing temperature explored keep the lamellae structure of pearlite as well as its metallographic and crystallographic texture unchanged. Though the residual stresses were not removed totally, their level of around 20 MPa after annealing is not significant. Then, these samples were deformed by tension and, again, residual stresses were determined

The results presented in Fig. 5 indicate that the characteristic small strain behavior observed in [11] is restored after annealing of the heavily drawn pearlite: the interphase stress increases in similar manner as for undrawn pearlite. Hence, the texture does not influence the internal stress considerably. More-



**Fig. 4.** Variation of interphase microstresses in ferrite and the reduction in cross-area with true strain of drawing.

over, as it is seen in Fig. 5, the interphase stress is some higher in the drawn and annealed pearlite than in undrawn pearlite with the similar interlamella spacing. This effect can be attributed to the polycrystalline structure of cementite lamellae after annealing [12,13], while before drawing the lamellae are single crystals.

#### 4. DISCUSSION

To discuss the behaviour of interphase stress, main peculiarities of the microstructure of heavily drawn pearlite should be outlined previously. The microstructure of the pearlite studied in the present work was examined earlier [8]. Before cold drawing, pearlite colonies exhibit fine lamellar microstructure with the thickness of ferrite and cementite layers 150...200 nm and 20...25 nm, respectively. Examination of the layer thickness after drawing showed that ferrite and cementite layers equally thinned. That agrees with the previously established fact that fine cementite is deformed plastically as well as ferrite [1,2]. Within a deformed pearlitic colony ferrite was shown to be subdivided to highly misoriented regions [8,14]. Dislocation density inside these regions is not too high. Instead of this, dislocations accumulate mainly at the interface. Cementite lamellae are distorted much harder than ferrite during drawing [12,13]. It is to be noted, that a lost of cementite layer integrity was observed, due to both a fragmentation of lamellae [1,2] and partial dissolution of cementite [13,15-17]. But the later effect is significant at very large true strains,  $\epsilon > 3$ . At the strains considered in the present investigation,  $\epsilon < 2$ , the cementite lamellae seem to keep their integrity over the most part of their extension [8,12].

Annealing of the pearlite drawn to a true strain up to 3 at temperatures around 400 °C results in the recovery of ferrite, and keeps lamellar morphology of pearlite [12,13]. At the same time, internal structure of cementite lamellar is recrystallized. For example, after drawing to  $\epsilon = 1.67$  and subsequent annealing at 420 °C the cementite lamellae have nanosized polycrystalline structure with the crystallite size about 20 nm [12].

Let us discuss now the decrease of interphase microstress during drawing observed in the present study. Factors that can influence such an evolution are: (i) the crystallographic texture development, alignment of colonies and lamellae inside the colonies (metallographic texture) [1], (ii) changing fine structure of ferrite and cementite. After annealing both the crystallographic and metallographic texture keep unchanged. Nevertheless, the result of

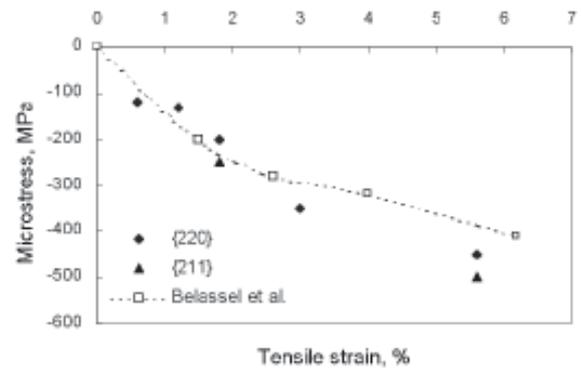


Fig. 5. Variation of interphase microstresses with tensile deformation of annealed specimens.

annealing is the return of the interphase microstress behaviour characteristic for undrawn pearlite. Since only limited recovery of ferrite phase occurs, it is reasonably to conclude that this is due to recrystallization of cementite. Therefore, the result of annealing experiment supports earlier suggestion [10] that the significant decrease of interphase microstress under cold drawing is due to significant transformation of the cementite lamellae substructure under severe plastic deformation.

#### 5. SUMMARY

1. Microstress appeared at early stage of cold drawing due to plastic interaction of the ferrite and cementite phases of lamellar pearlite, called the interphase stress, gradually decrease during further drawing.
2. The small strains behavior, that is the rapid increase of interphase stress with plastic strain, is restored by way of annealing which keeps crystallographic and metallographic texture of heavily drawn pearlite.
3. The annealing experiment supports the suggestion that the significant decrease of interphase microstress is due to evolution of the cementite substructure in the course of drawing.

#### REFERENCES

- [1] M. Zelin // *Acta Mater.* **50** (2002) 4431.
- [2] G.Langford // *Metall Trans.* **8A** (1977) 861.
- [3] V.S. Zolotovskiy, In: *Mechanical testing and properties of metals* (Moscow, Metallurgia, 1974) p. 159, in Russian.
- [4] D.M. Vasiliev, N.Yu. Zolotovskiy, Yu.F. Titovets, B. Buchmayr and G. Hampejs //

- Trans. St.-Petersburg Academy of Sciences for Strength Problems* **1** (1997) 233.
- [5] K. Van Acker, P. Van Houtte and E. Aernoudt, In: *Proc. 4<sup>th</sup> Int. Conf. on Residual Stresses* (SEM, 1994) p. 402.
- [6] N. Yu. Zolotarevsky, N. Yu. Krivonosova and S. A. Ivanov // *Industrial Laboratory* **12** (1994) 27.
- [7] N. Yu. Zolotarevsky and N. Yu. Krivonosova // *Mater. Sci. Eng.* **A205** (1996) 239.
- [8] E. V. Nesterova, V. V. Rybin and N. Yu. Zolotarevsky // *Fiz. Met. Metalloved* **89** (2000) 47, in Russian.
- [9] W. F. Hosford Jr // *Trans. Metal. Soc. AIME* **230** (1964) 12.
- [10] N. Yu. Zolotarevsky, D. M. Vasiliev and Yu. F. Titovets // *Problems of Materials Science* **33** (2003) 104.
- [11] M. Belassel, J. L. Lebrun and J. P. Bettembourg, In: *Residual Stresses; Proc. European Conf. on Residual Stresses*, ed. by V. Hauk *et al.* (DGM, 1993) p. 779.
- [12] K. Maki, H. Yaguchi, M. Kaisio, N. Ibaraki, Y. Miyamoto and Y. Oki // *Scripta Mater.* **37** (1997) 1753.
- [13] J. Languillaume, G. Kapelski and B. Baudalet // *Acta Mater.* **45** (1997) 1201.
- [14] E. V. Nesterova, N. Yu. Zolotarevsky, V. V. Rybin and Yu. F. Titovets // *Problems of Materials Science* **33** (2003) 250.
- [15] H. G. Read, W. T. Reynolds Jr., K. Hono and T. Tarui // *Scripta Mater.* **37** (1997) 1221.
- [16] W. J. Nam, Ch. M. Bae, S. J. Oh and S. Kwon // *Scripta Mater.* **42** (2000) 457.
- [17] V. G. Gavriljuk // *Scripta Mater.* **45** (2001) 1469.