

**RESIDUAL STRESSES IN SURFACE LAYER OF M2HSS STEEL  
AFTER CONVENTIONAL AND LOW PRESSURE  
("NITROVAC'79") NITRIDING PROCESSES.**

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**Summary**

*The distribution of residual stresses in the surface layer of M2HSS steel after conventional and low pressure ("NITROVAC'79") nitriding is reported and discussed in this paper.*

*The stresses depend on the structure of the nitrided layer, which results from the applied nitriding parameters. The optimum structure has been obtained after low pressure nitriding at ammonium partial pressure  $p=20$  hPa.*

*Cyclic annealing after nitriding at temperatures below nitriding temperatures doesn't cause any significant changes in residual stresses' distribution. Annealing temperatures higher than resorted at nitriding, causes a well-marked increase of residual stresses in surface layer of the steel.*

**1. Introduction**

The most surface treatments are aimed to produce a specified state of residual stresses which have a very significant influence on mechanical and tribological properties of tools and machine parts. For that reason, to define the dependence between static and dynamic strength indexes is a very important problem both from the scientific point of view and applicational one. Technologies that essentially change the state of stresses are the thermochemical ones, especially nitriding.

The papers that have been published hitherto [1÷5] concentrated on investigations of residual stresses obtained in conventional gas nitriding processes, mainly. Similar studies of residual stresses in the  $\gamma'$  layer after ionic nitriding are given in the paper [6].

The wide in a scale studies on entirety problems connected with low pressure nitriding ("NITROVAC 79") [7] are conducted in the Institute of Material Engineering and Chipless Technologies of Technical University of Łódź. The method is widely applied in manufacturing of various machinery and devices' elements as well as multiedge cutting tools. Low pressure nitriding allows to form the structure of the surface layer optionally [8]. It makes the fundamental difference between this type of nitriding and conventional one because the porous and brittle layer of  $\epsilon + \gamma'$  nitrides obtained in conventional process should be removed by rather expensive, final grinding.



The investigations that refer to the comparison of residual stresses distribution in surface layers of M2HSS steel after conventional and low pressure nitriding, are presented in this paper. Taking into account that the nitriding processes are applied for cutting tools working at periodically changing temperatures, a trial to estimate the influence of temperature on the residual stresses has been undertaken.

## 2. Experimental

### 2.1. Materials investigated

The specimens in form of the plates (120x20x2,5 mm) were prepared from M2HSS steel. The specimens were exposed to the following heat treatment:

- austenizing temperature: 1483K
- cooling media: oil
- tempering temperature: 843K
- tempering time: 1h

The hardness, after bulk heat treatment, has been assessed by Rockwell method in C-scale. The average result for all specimens was  $63 \pm 0,5 \text{HRC}$ .

The low pressure nitriding has been realised at partial ammonium pressures:  $p_1=20 \text{hPa}$  and  $p_2=180 \text{hPa}$ , for two series of samples, the third one underwent conventional nitriding. The parameters of nitriding processes were: temperature-833K, time-6h, dissociation degree  $\sim 30\%$ , in all mentioned above cases. The part of low pressure nitrided samples (process pressure  $p=20 \text{hPa}$ ) was subjected, additionally, to cyclic vacuum annealing (5x30min with periodical cooling to 373K) at temperatures: 673K, 773K, and 873K.

The structure, thickness of diffusion zones and residual stresses were investigated in every group of the specimens.

### 2.2. Structural investigations

Phase composition of the obtained diffusive layers was investigated on D500 Siemens diffractometer with use of  $\text{CoK}_\alpha$  radiation. The results are given on Fig. 1.

The metallographic structure was examined either. The results are given in Table 1 and on Fig. 2.

### 2.3. Microhardness test.

Microhardness tests were made by using Vickers test with applied load of 1N on SOPELEM apparatus. The results (average from 5 measurements) are given on Fig. 3.



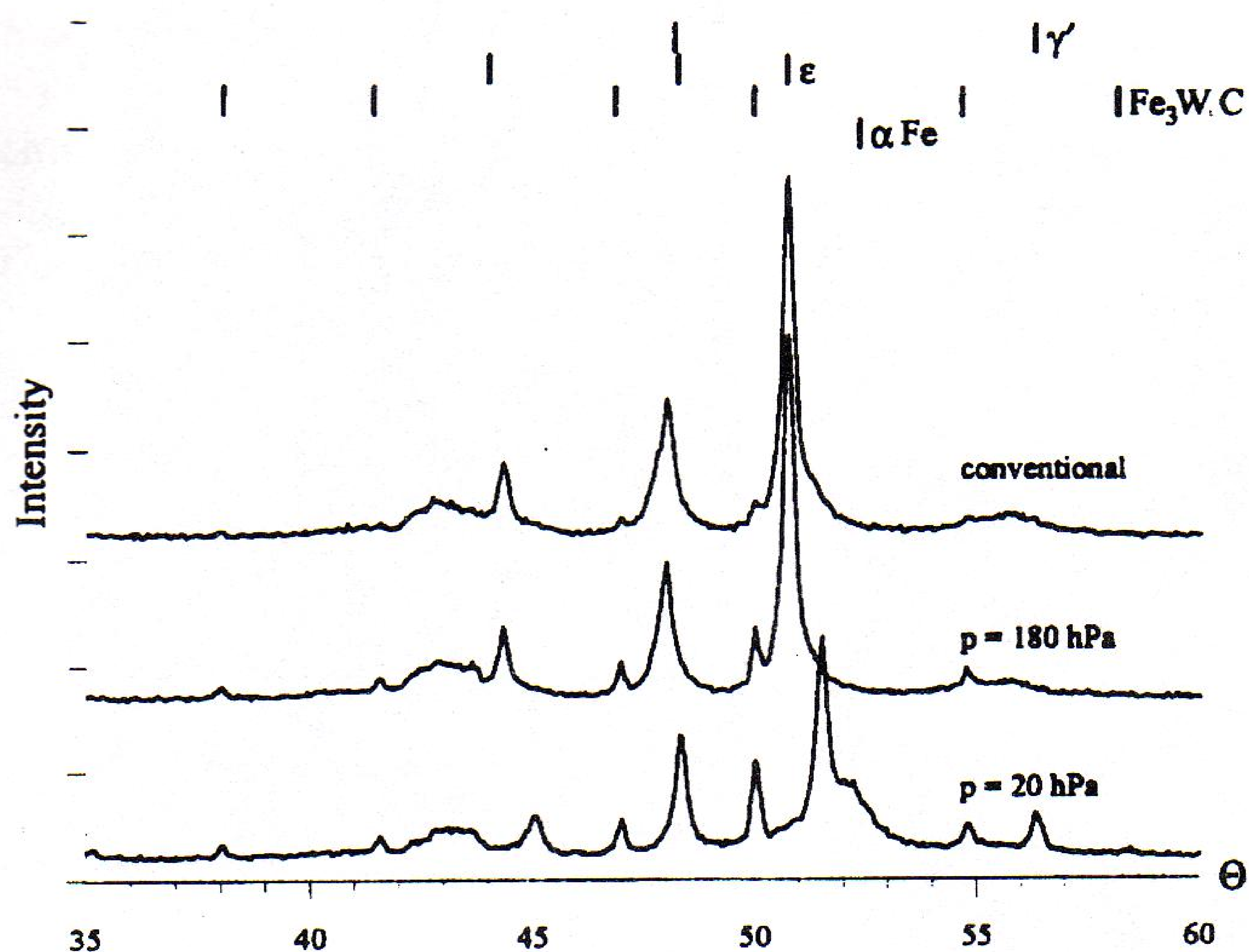
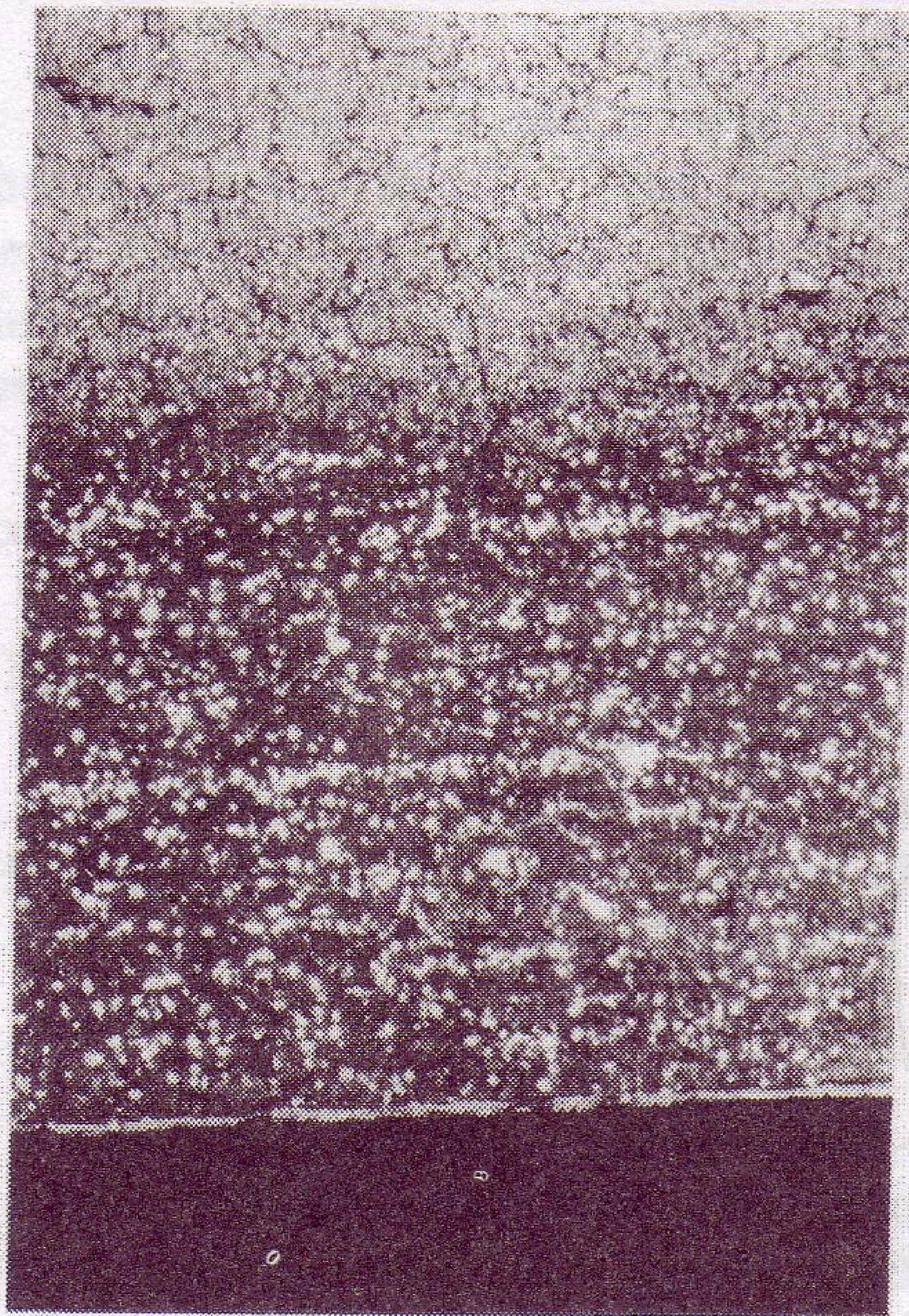
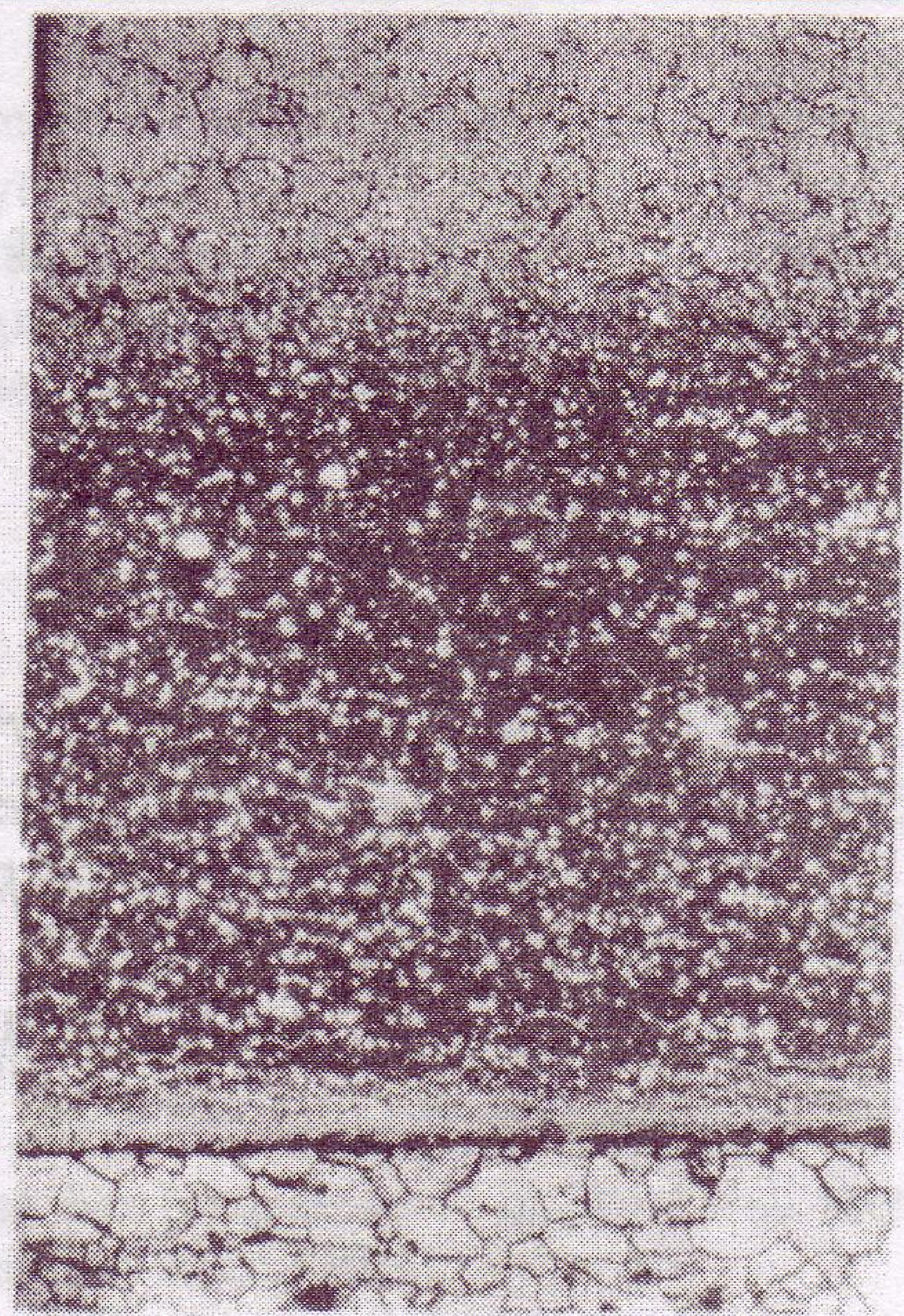
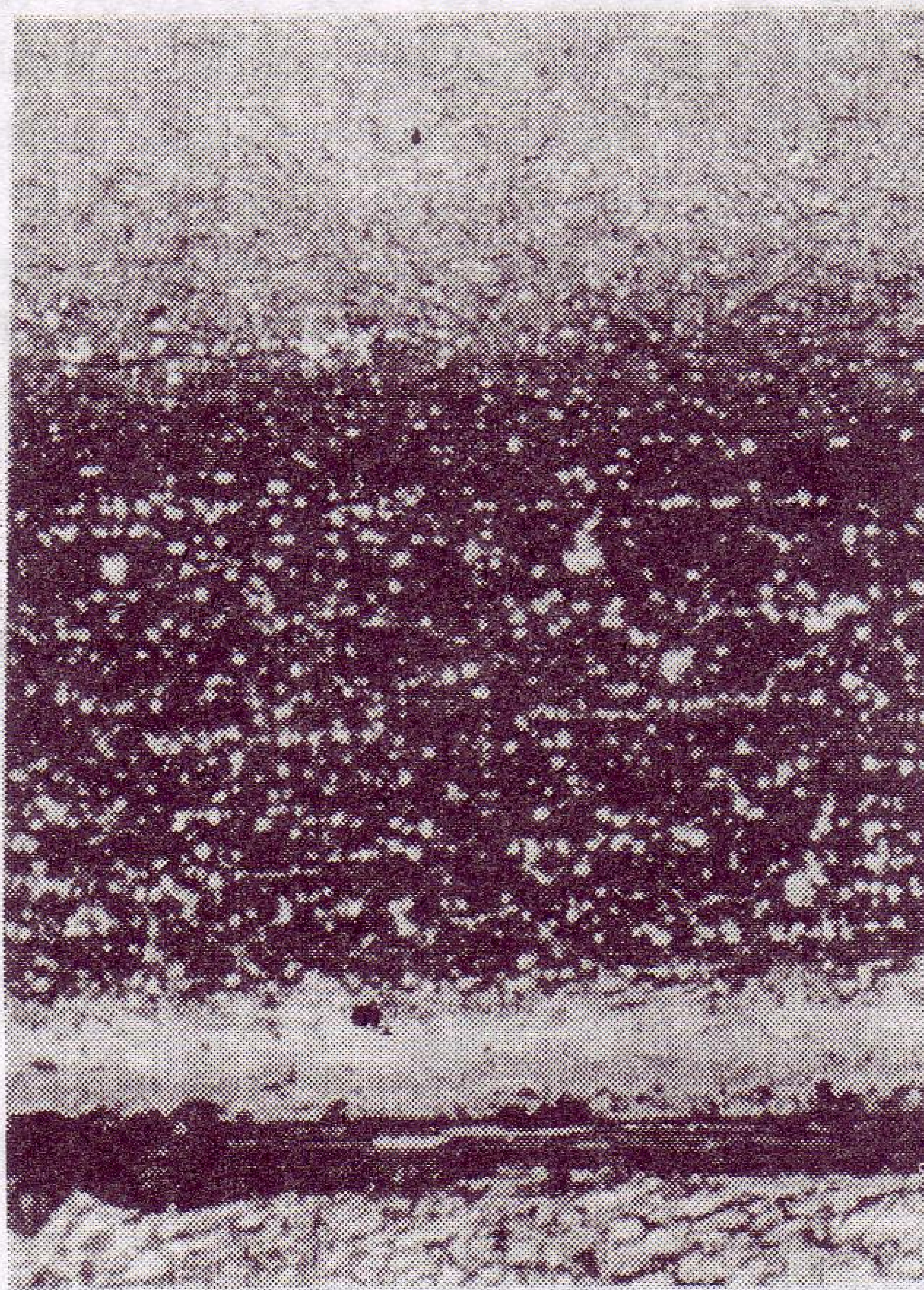


Fig. 1. Diffractogram from the surfaces of nitrided specimens

Type of nitriding	Nitriding time	Nitriding temperature	Type and thickness of the layer		
			Inner nitriding zone	$\gamma$	$\epsilon + \gamma$
NITROVAC'79 p=20hPa	6 hours	843K	~100 $\mu$ m	2+3 $\mu$ m	-
NITROVAC'79 p=180hPa	6 hours	843K	~125 $\mu$ m	-	~8 $\mu$ m
Conventional	6 hours	843K	~100 $\mu$ m	-	~17 $\mu$ m

Table 1. The thickness of nitrides' layers and inner nitriding zone



2a)  $p = 20 \text{ hPa}$ 2b)  $p = 180 \text{ hPa}$ 

2c) conventional

Fig. 2. Microstructure of former heat treated and nitrided M2HSS steel  
- low pressure (2a and 2b),  
- - conventional 2c  
Etched                      magnification: 400x



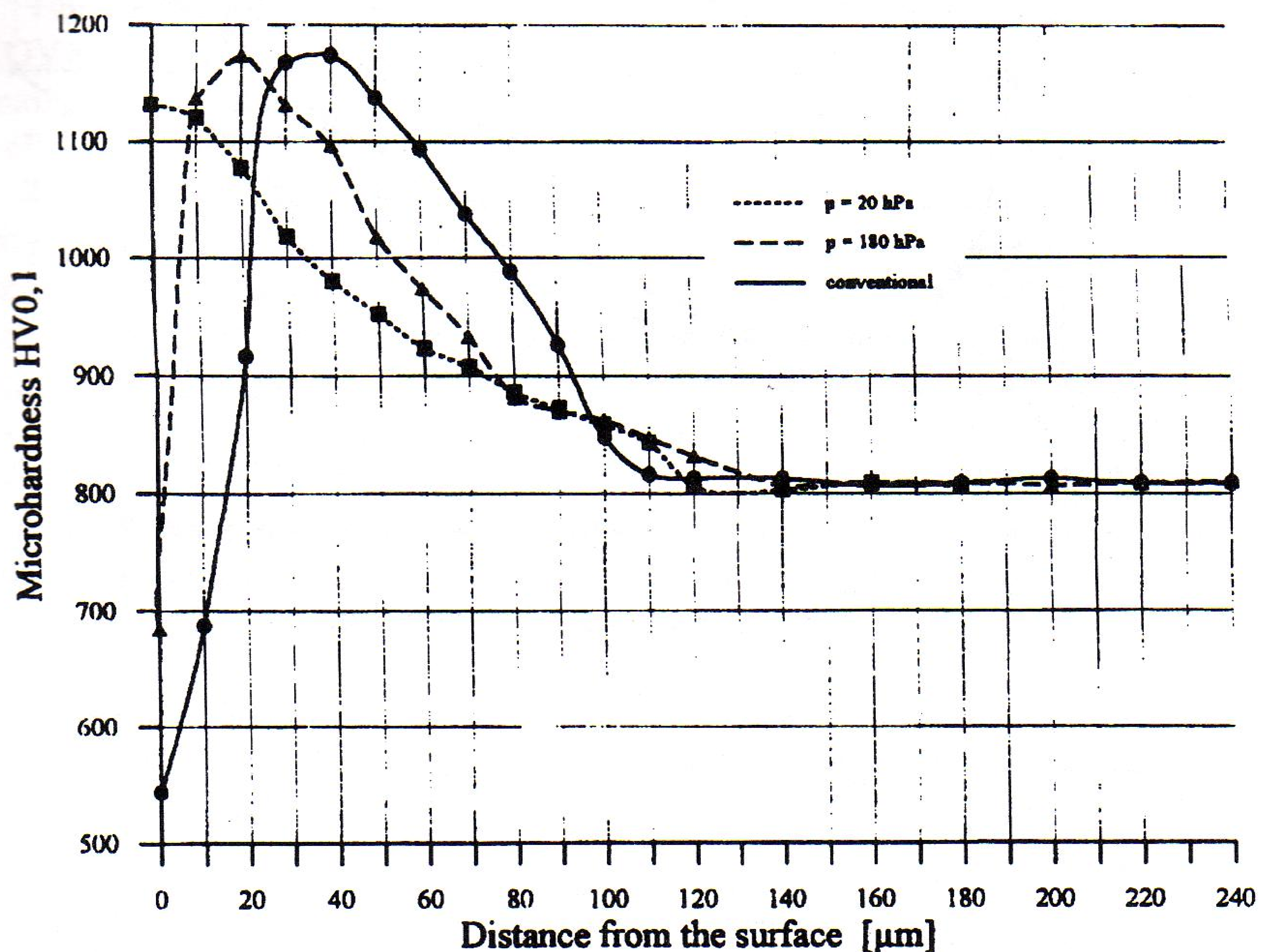


Fig. 3. Distribution of average microhardness in surface layer of former vacuum and conventional nitrided M2HSS steel

#### 2.4. Residual stresses examination.

Three specimens from each series underwent the Waisman-Phillips test [9]. In accordance to the author of paper [10] this method, in much better way, gives the distribution of residual stresses in surface layer, then a commonly use X-ray analysis ( $\sin^2\psi$ ). Waisman-Phillips method is based on measuring of the deflection arrow of the flat specimens that deform during releasing the stresses in consequence of electroetching of consecutive material layers. Young modulus of particular nitrided layers ( $\epsilon$  and  $\gamma'$ ) and substrate, should be known to calculate the residual stresses. These values are given in paper [11] and they are  $E_\epsilon=181,5\text{GPa}$ ,  $E_{\gamma'}=211,0\text{GPa}$ ,  $E_\alpha=206,0\text{GPa}$ , respectively.

The results of the investigations are given on Fig. 4 and 5.



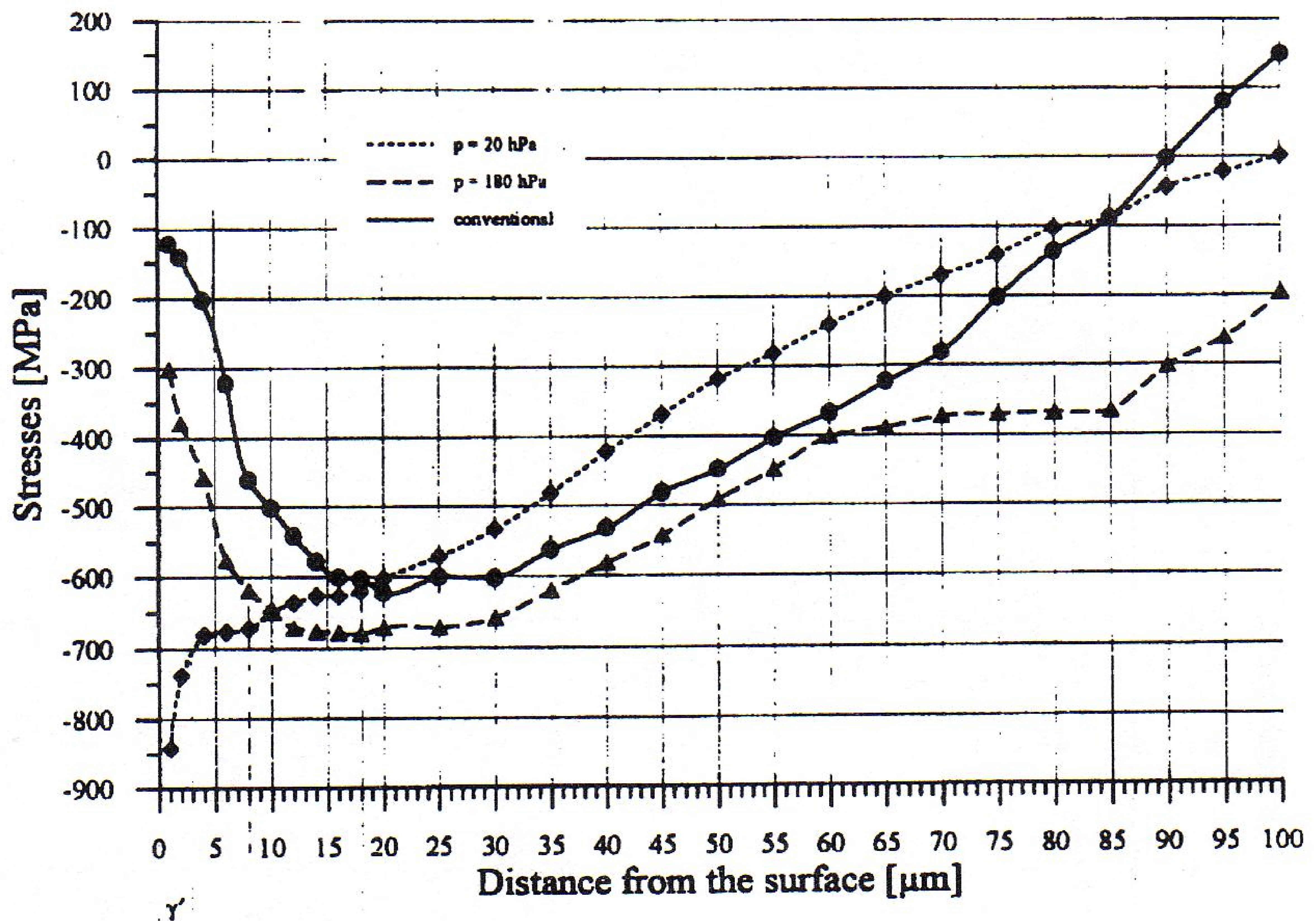


Fig 4. Distribution of residual stresses in surface layer of former low pressure and conventional nitrided M2HSS steel.

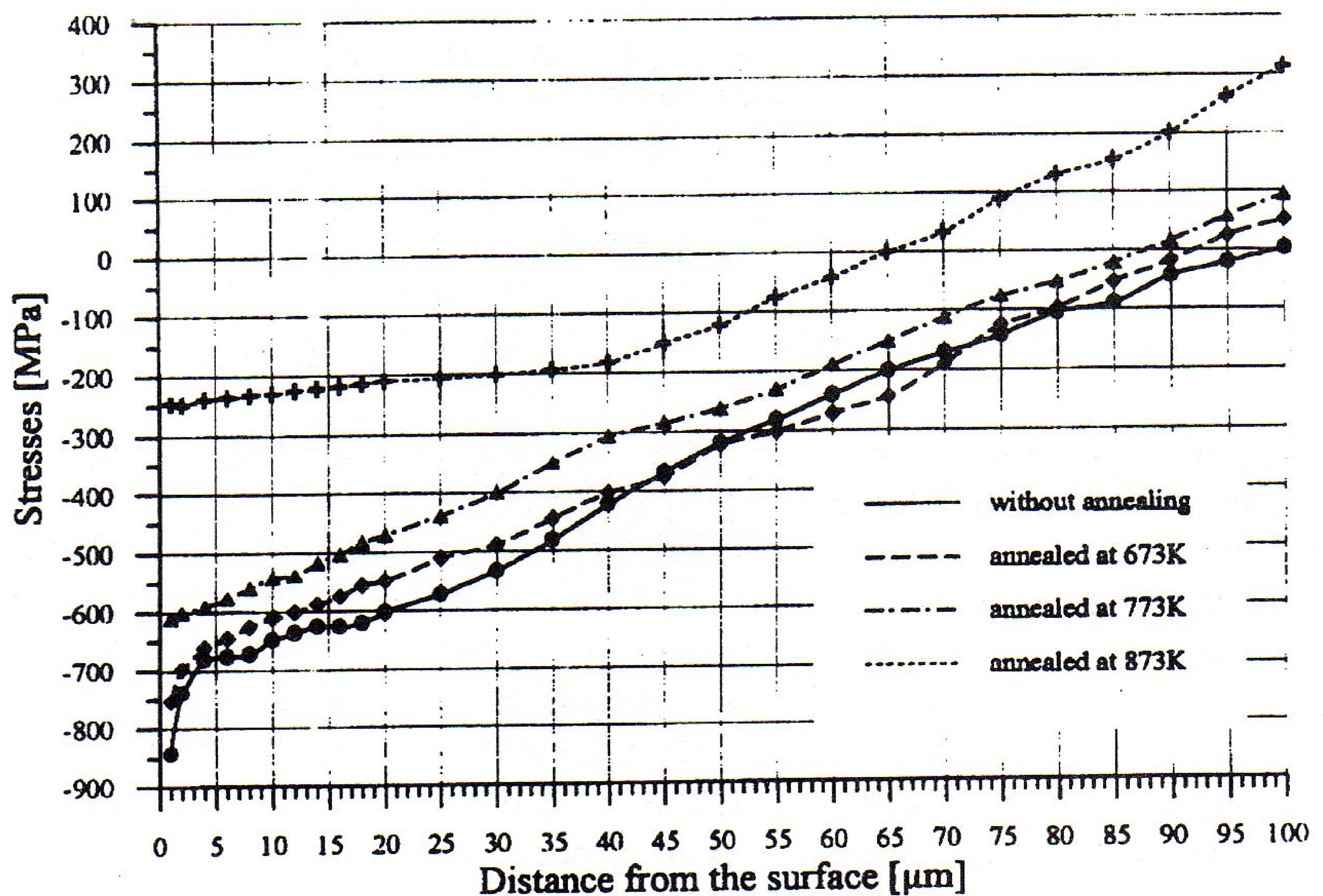


Fig. 5. Distribution of residual stresses in surface layer of former vacuum nitrided M2HSS steel at  $p=20\text{hPa}$ , cyclically annealed at temperatures 673K, 773K and 873K.



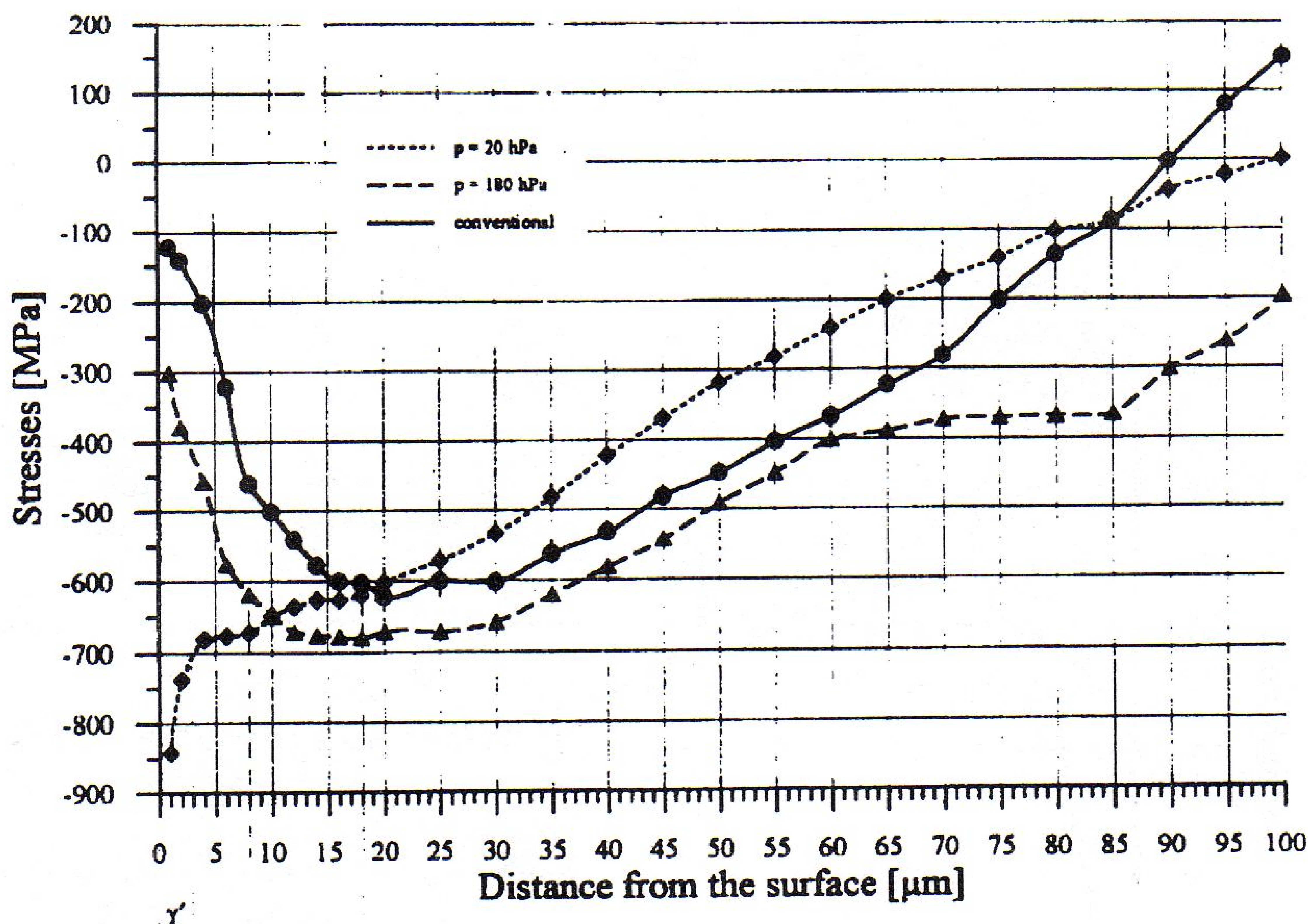


Fig 4. Distribution of residual stresses in surface layer of former low pressure and conventional nitrided M2HSS steel.

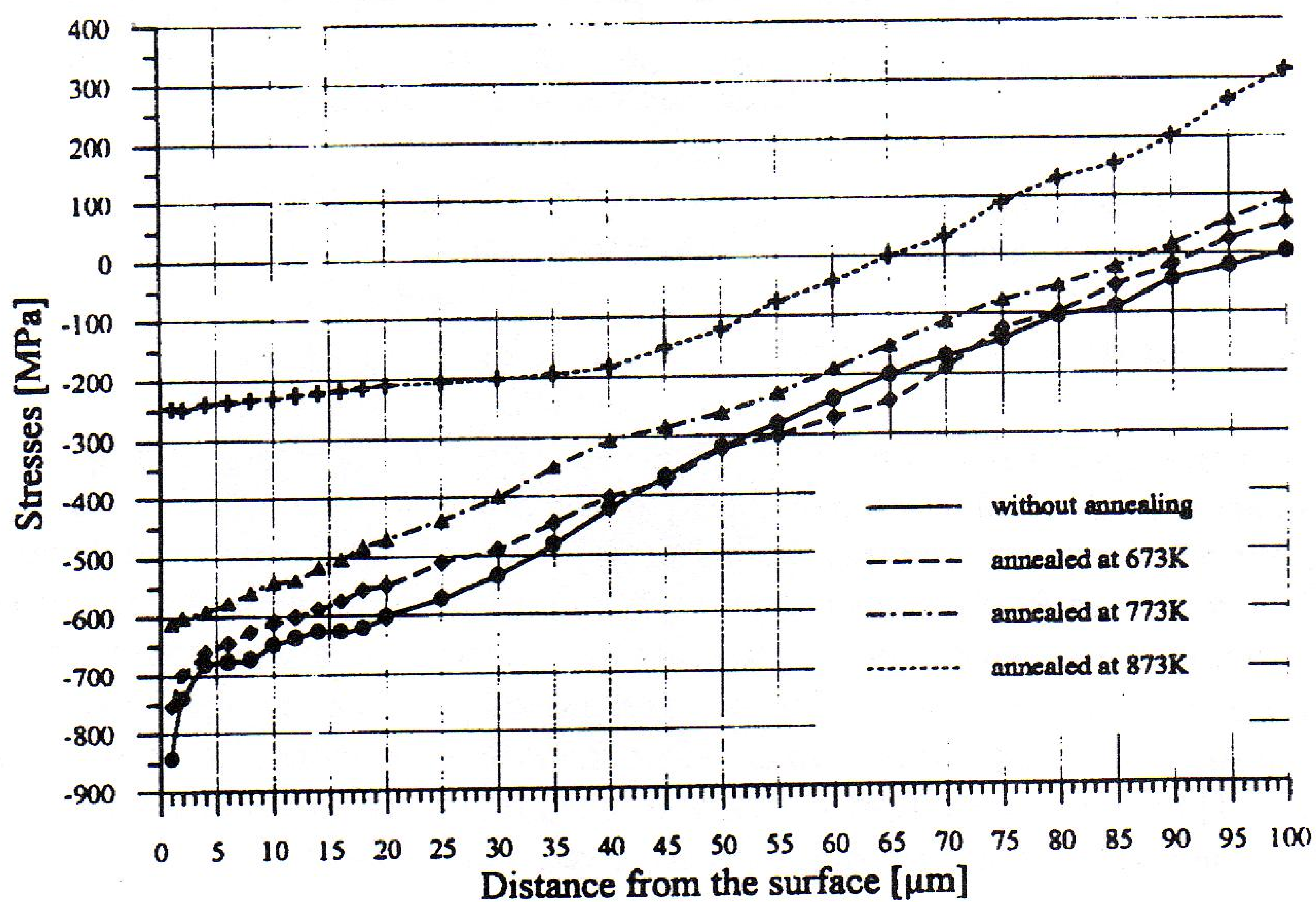


Fig. 5. Distribution of residual stresses in surface layer of former vacuum nitrided M2HSS steel at  $p=20\text{hPa}$ , cyclically annealed at temperatures 673K, 773K and 873K.



### 3. Discussion.

The results of microstructural investigations show that the vacuum nitriding NITROVAC'79 allows, similarly as ionic nitriding, forming the surface structure, optionally. The surface layers without porous and brittle nitrides (which are in most cases objectionable) can be obtained at some parameters of low pressure nitriding.

The change of the ratio of nitrogen atoms amount adsorbed on the surface, to the quantity of their absorption (diffusion into the material) makes possible the structure forming of surface layer. It can be obtained by changing the partial pressure of nitrogen. In conventional nitriding the adsorption of atomic nitrogen is too large, in the rate to absorptional ability of the material, what causes the accumulation of the overbound atomic nitrogen on the surface, and in consequence, the creation of the thick nitrides' layer ( $\epsilon+\gamma'$ ).

The distribution of average microhardness in nitrided layers, for particular treatment's variants seems to be very interesting. The ammonia gas partial pressure increase gives the inconsiderable increase of maximum hardness (from 1130HV to 1180HV), but it occurs deeper and deeper in the structure.

Analysing the Fig. 4 (distribution of residual stresses) it can be well-marked that the nitriding pressures have a significant influence on the stresses character and values. Maximum compressive stresses (-842MPa) occurred at  $p=20\text{hPa}$ , near the surface of the specimen. The course of residual stresses distribution curve changes in the range of  $20\mu\text{m}$ , at pressure  $p=180\text{hPa}$ . The value of compressive stresses is much lower in the first case, and its number is -301MPa, close by the surface. This value increases and reaches the maximum (680MPa) at distance  $18\mu\text{m}$ , with moderation of deflection from the surface. The distribution curve of residual stresses has the similar character, in case of the conventional nitriding, but the value of stresses is significantly lower: -122MPa. The maximum value of compressive stresses (603MPa) is obtained at depth  $18\mu\text{m}$ .

The conclusion is that the structural formations of nitrided layers determine the values and course of residual stresses. At partial ammonium pressure  $p=20\text{hPa}$  the rigid layer of  $\gamma'$  nitrides ( $2-3\mu\text{m}$ ), and below the inner nitriding zone reaching  $100\mu\text{m}$  (Fig. 2a), have been obtained, in case of low pressure nitriding. In case of ammonium partial pressure  $p=180\text{hPa}$  the "white layer" of nitrides  $\epsilon+\gamma'$  is obtained (about  $8\mu\text{m}$ ) and below we have the inner nitriding zone, about  $125\mu\text{m}$  (Fig. 2b). The  $\epsilon$  nitrides in the surface layer, significantly decreases the maximum value of compressive stresses (in comparison to first treatment variant). This phenomenon can be better observed in case of conventional nitriding when the "white layer" of  $\epsilon+\gamma'$  nitrides (about  $17\mu\text{m}$ ) was obtained (Fig. 2c). The scanning microscope investigations have shown that this layer is much more porous then in case of second treatment variant.

So we can conclude that the nitrides  $\epsilon+\gamma'$  layer obtained at specified treatment parameters, significantly decreases the value of compressive stresses on the specimens' surface. It was satisfied, that with the increase of ammonium partial pressure, in range the white layer occurs, the porosity of this layer increases. This has the significant influence on stresses level on the specimen surface.



The low pressure nitriding NITROVAC'79 of M2HSS steel, at partial ammonium pressure  $p=20\text{hPa}$ , assures optimal microstructure and residual stresses distribution in case when the decisive exploitation factors are unit thrusts and frictional wear what results from above.

Cyclic annealing at temperatures 673K and 773K doesn't cause the significant changes in residual stresses distribution of nitrided specimens both in the structure and chemical composition. The significant change of residual stresses occurred after the cyclic annealing at 873K (the temperature 40K higher then nitriding one). A significant decrease of compressive stresses on the surface to  $-250\text{MPa}$  (it is about  $600\text{MPa}$  in comparison to steel after nitriding), than occurs. It can be supposed that this is the result of very intensive diffusion processes between  $\gamma'$  phase and the substrate as in the substrate itself, what leads to the stresses' relaxation. The precipitation of nitrides phases in the substrate occurs and the coefficients of line thermal expansion of phases  $\gamma'$  and  $\alpha$  reach approximate values.

#### 4. Conclusions

NITROVAC'79 low pressure nitriding process allows forming the nitrided layers on M2HSS steel, optionally.

The residual stresses distribution in the nitrided layer depends on its structural formation, and indirectly, on process parameters.

Optimal structure was obtained at ammonium partial pressure  $p=20\text{hPa}$ .

Optimal structure, mentioned above, consists of very thin ( $2\text{-}3\mu\text{m}$ ) sublayer of rigid  $\gamma'$  nitride and inner nitriding layer.

Cyclic annealing, after the low pressure nitriding, in temperature under the nitriding one, doesn't cause any significant changes in the residual stresses distribution. Annealing at temperature higher then nitriding one leads to significant decrease of residual stresses in surface layers.

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