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DEFORMATION ZONE GEOMETRY AND TEXTURE GRADIENTS IN COLD-ROLLED NIOBIUM†

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Abstract: Several niobium plates were cold-rolled at room temperature to a total reduction of 60% maintaining the geometry of the zone of deformation constant for each plate. Pole figures were obtained by means of the Schulz x-ray reflection technique from various depths in the thickness direction for plates rolled with different Δ, the ratio of the mean height of the deformation zone to its contact length. Severe texture gradients were noted and characterized for Δ > 1; a modified texture different from the normal texture was observed at intermediate through-the-thickness locations. Both lateral widening and microhardness gradients were also in evidence for this case. No previously proposed theoretical explanation could account for these results.

INTRODUCTION

The cold-working of metals and alloys by rolling causes grain shape change and grain crystallographic reorientation to preferred orientations which can result

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in a highly textured product. Attempts to theoretically understand and thus to predict texture development idealize the stress-state of the rolling process, assume plane-strain deformation and further consider that the plastic deformation of the work-piece occurs homogeneously, i.e. a transverse vertical section remains planar and vertical as it passes through the roll gap. In this latter regard experimental investigations such as those by Tarnovskii *et al.*[1] have demonstrated, however, that under certain circumstances, the distortion of coordinate grids embedded within the zone of plastic deformation differed substantially from one layer to another in the through-the-thickness direction. Viewed with this perspective it might be considered that the occurrence of layer-to-layer variations in texture reported in a number of studies[2-6,9] is an indirect manifestation of accumulated inhomogeneous flow behavior during rolling.

At least two factors are thought to promote significant inhomogeneous flow. First, severe frictional or shearing forces between the work-piece and rolls can cause surface-to-center texture variations. Often a unique surface texture appears which is dramatically different from the interior texture. The surface texture generally evolves after large amounts of accumulated deformation utilizing heavy reductions per pass with inadequately lubricated rolls. The surface in rolling of BCC metals[5-7] can consist of the same preferred orientations that are observed in torsion or pure shear deformation experiments[8] with the rolling direction corresponding to the shear direction and the rolling plane to the shear plane.

A second factor which can contribute to manifestations of inhomogeneous rolling is the geometry of the zone of deformation. The deformation zone in rolling is schematically depicted in Figure 1, and its geometry can be characterized by a shape factor, Δ, defined as

$$\Delta = \frac{t_m}{\ell} \cong \frac{t_a + t_b}{2\sqrt{R(t_a - t_b)}} , \tag{1}$$

where t_m/ℓ is approximately the mean thickness-to-contact length ratio of the plastic zone filling the roll gap; R is the roll radius and t_a and t_b are the entrance and exit thicknesses of the rolled work-piece.* A relation-

*The shape factor defined here is the reciprocal of the one set forth by Tarnovskii *et al.*[1] and used in our previous paper.[5]

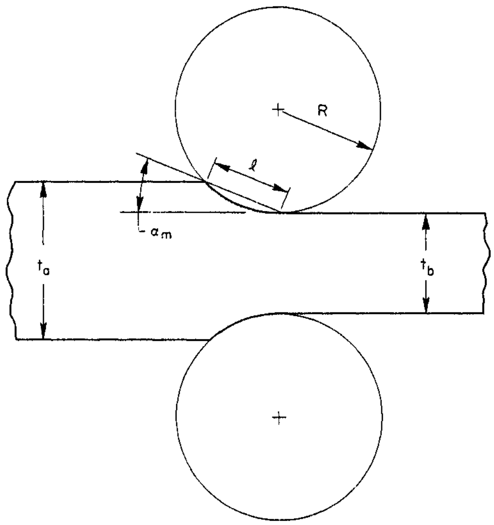

Figure 1. Deformation zone geometry for the rolling of plates.

ship between deformation zone geometry and texture inhomogeneity was tentatively proposed by Vandermeer and Ogle[5] to rationalize bulk texture gradients observed after moderate amounts of rolling of niobium specimens. More recently Mathur and Backofen[10] demonstrated a connection between Δ and the uniformity and nature of textures in cold drawn and recrystallized aluminum-killed steel strip.

Since there exists a paucity of information regarding this second factor and its contribution to non-homogeneous deformation, the primary purpose of the present work was specifically to investigate the effects of deformation-zone geometry on rolling texture homogeneity or lack thereof in polycrystalline niobium. To that end, annealed niobium plates were rolled to approximately 60% reduction in thickness maintaining the deformation-zone-shape similar for each and every pass through the rolling mill. This paper describes the nature of the textural gradients which resulted for different zone geometries. Some texture variations were considerably more drastic than expected.

EXPERIMENTAL

The niobium used in this study was from the same electron-beam-melted billet stock used in the previous experiments.[4,5] The plate and bar specimens to be rolled were machined from this billet stock after it was repeatedly fabricated and annealed to produce a reasonably fine-grained material with minimal texture.[4] Two size categories of specimens were used. Most were plates nominally 10-cm wide and 1-cm thick. Others were bar-shaped approximately 3.2-cm wide by either 1.1-cm or 2-cm thick.

Rolling was accomplished at room temperature between 20-cm diameter rolls in a mechanically operated, two-high mill manufactured by Ruesch. Prior to rolling a light film of machine oil was swabbed on the rolls. The work-piece was hand-fed into the roll gap and reversed end-for-end after each pass. The shape factor Δ was maintained constant for each specimen. Shape factors of either 1/2, 2/3, 1, 4/3, 2, or 4 were employed to reduce the specimens to an accumulated reduction in thickness of nominally 60%.

In order to roll at constant Δ with the same rolling mill, it was necessary to adjust the draft to a specific value with each pass through the mill. A master table listing the thickness, t_a, and the necessary draft, $t_a - t_b$ required to roll that thickness of work-piece to maintain a particular Δ was calculated from Equation (1). To ensure that the actual draft was close to the one sought for, a trial and error calibration of the mill by rolling dummy blocks was carried out. The actual shape factors were usually within 2% of the nominal (sought for) value except when Δ was less than 1 and then the factors were always within 8%. It should be remarked that when rolling under these conditions, the equivalent die angle, α_m in Figure 1, does not remain constant but decreases slightly from one pass to the next. Table I lists some of the pertinent rolling parameters for four of the specimens whose textures were examined in detail.

TABLE I

Rolling Parameters

Δ Shape Factor	Total Reduction (%)	Total No. of Passes	Initial Width/ Thickness Ratio	α_m First Pass	α_m Last Pass	Longitudinal Curvature (cm)
1/2	62	4	10	4°17'	2°37'	∿ ∞
1	62	16	10	3°16'	1°2'	20.7
2	62	63	10	1°48'	34'	14.6
4	59	71	1.6	2°19'	46'	Not measured

The samples for texture determinations were 2.5 by 2.5-cm wafers machined from the center of the rolled plates. To obtain pole figures at specific depths, material was removed in a systematic way from the surfaces parallel to the rolling plane by carefully controlled chemical polishing in a solution of 70% by volume of nitric acid and 30% hydrofluoric acid.

The (110) and (200) pole figures were determined by the Schulz x-ray reflection technique using CuK_α radiation. A PDP-8 computer commanded the scanning movements of a Norelco texture goniometer and allowed the diffracted intensities to be automatically recorded. The measured intensities were corrected for background, normalized to data collected from a random niobium powder specimen and converted to pole figures using a program written by Love[11] and the IBM 360 computer.

RESULTS

Some of the rolled plates developed a slight overall longitudinal curvature after the first passes through the rolling mill. This curvature was perpetrated and intensified by additional rolling. As Table I shows, specimens rolled with high Δ (multitudinous passes) became more curved than those rolled at lower shape factors (fewer passes). An extreme example of the longitudinal curvature along with inhomogeneous lateral widening is shown in Figure 2 for a bar specimen rolled with $\Delta = 4$. The theoretical and experimental investigation of Dewhurst, Collins and Johnson[12] would suggest that the cause of this curvature may be related to small differences in either angular velocity or roll diameter or both, between the top and bottom rolls of the mill. Since the plates were not turned over after each pass these small differences "acted" cumulatively and the curvature increased as deformation continued. The consequences of these rolling procedures made it appear that the top and bottom surfaces deformed differently. Indeed, the neutral axis no longer corresponded to the midplane of the plate and the textures were not symmetrical with respect to the midplane. Thus the expected "double-barrel" shape did not develop in high Δ specimens. For low Δ specimens (high drafts) the longitudinal curvature was almost nonexistent and the texture results indicated that the neutral axis and specimen midplane nearly coincided.

The texture results and other observations may be described and discussed most conveniently by grouping the specimens into two classes according to shape factor. The first group, characterized by only mildly depth-dependent texture variations, were rolled with shape factor $\Delta \leq 1$. Figures 3 and 4 illustrate the nature of these variations for $\Delta = 1$ and 1/2, respectively. These figures compare the distribution of (110) poles near the

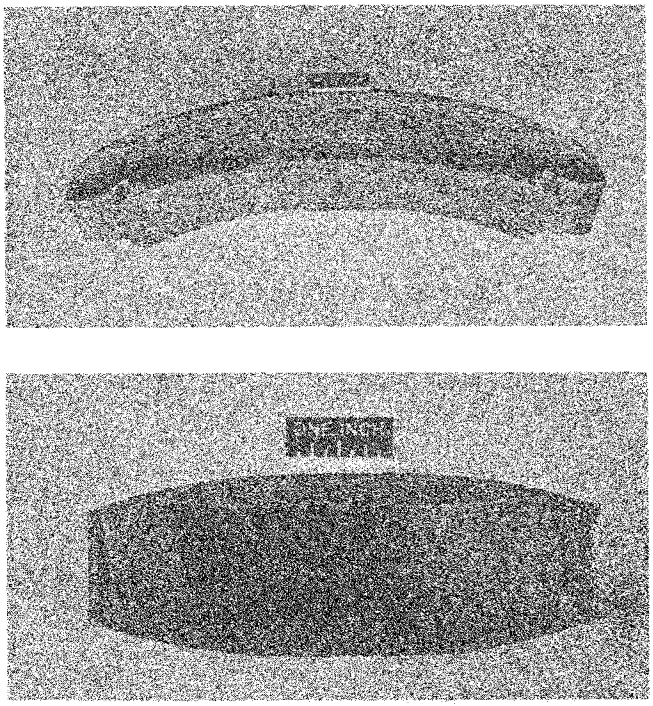

Figure 2. Side view (upper) and bottom view (lower) of a niobium bar rolled 59% with shape factor, Δ = 4.

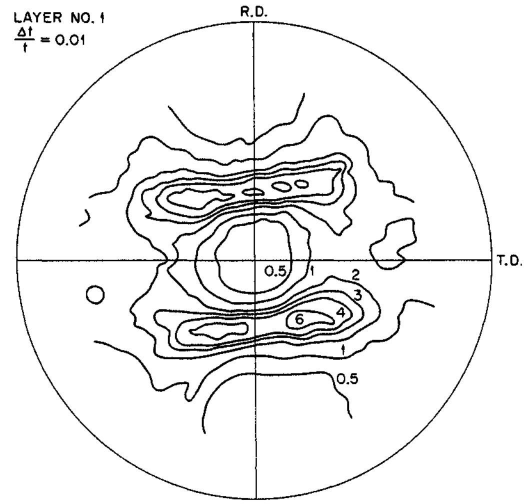

Figure 3a. (110) pole figure from the top surface of a niobium plate rolled 62% with Δ = 1.

surface of the rolled plate (a) with that at the midplane (b). It is evident that the texture was most nearly uniform when Δ = 1.

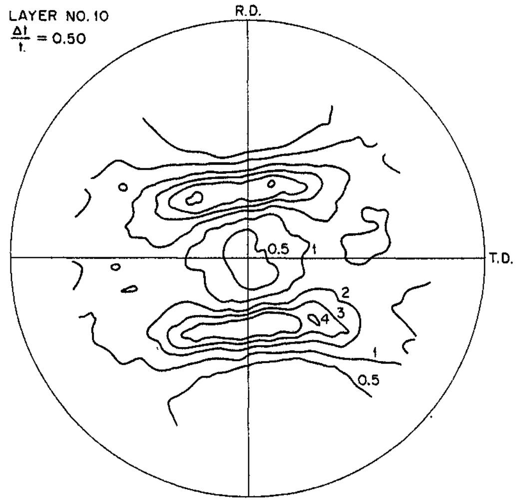

Figure 3b. (110) pole figure from the midplane of a niobium plate rolled 62% with Δ = 1.

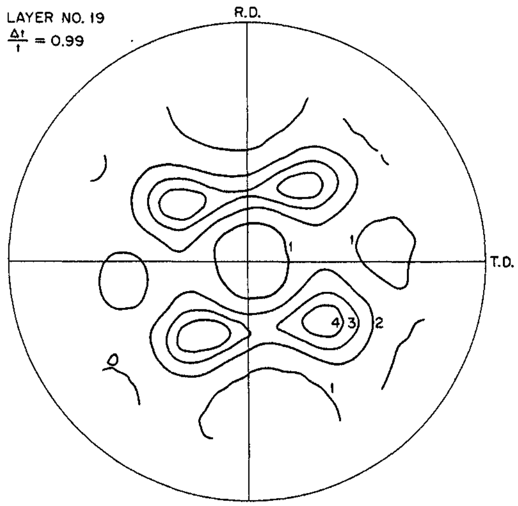

Figure 4a. (110) pole figure from the bottom surface of a niobium plate rolled 62% with Δ = 1/2.

The texture that developed in this group of specimens was in general agreement with previous findings for moderately rolled niobium[4] and other body-centered cubic (bcc) metals.[14] It may be described in terms of two types of continuous orientation spreads: For example, A-orientations extending from $(001)[\bar{1}10]$ to $(112)[\bar{1}10]$ and B-orientations ranging from $(111)[\bar{1}2\bar{1}]$ to $(112)[\bar{1}10]$. For a complete description see reference 4. Alternate descriptions of the B-orientations have been proposed by

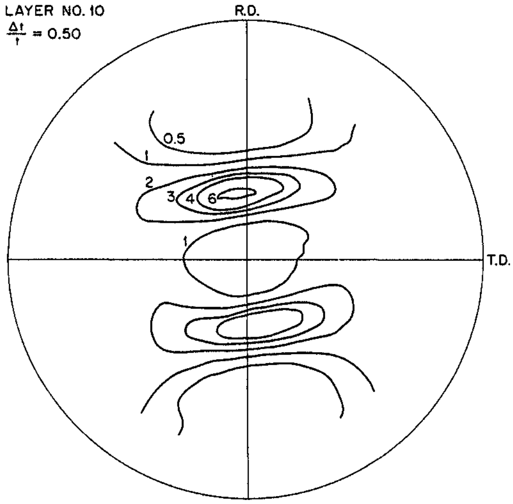

Figure 4b. (110) pole figure from the midplane of a niobium plate rolled 62% with Δ = 1/2.

others but it is not our purpose here to argue the merits or subtlety of each. A comparison of these has been presented previously.[4]

The nature of the weak textural inhomogeneities noted for the Δ = 1/2 specimen, is shown qualitatively with the data presented in Figure 5. The upper curve plots the

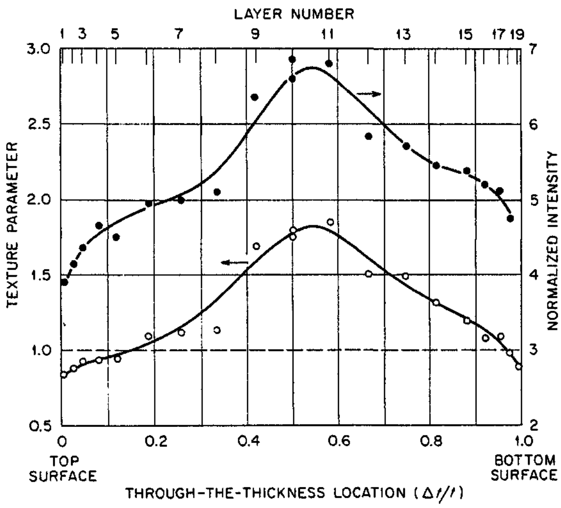

Figure 5. Depth-dependence of texture in a niobium plate rolled 62% with Δ = 1/2. Filled circles: normalized maximum intensity along ND to RD radius of pole figure. Open circles: texture parameter sensitive to amount of (112)[$\bar{1}$10] component (see text for definition).

maximum normalized intensity as measured along the normal direction (ND) to rolling direction (RD) radius of the pole figure versus through-the-thickness location. The lower curve depicts the variation of a parameter which is the ratio of this above mentioned intensity divided by the normalized intensity located at the (001)[$\bar{1}$10] ideal orientation, as a function of the same thickness variable. A rationalization of these data consistent with both (110) and (200) pole figures suggested that the ideal (112)[$\bar{1}$10] type orientations tended to be lacking in the surface and near-surface regions but were reasonably prominent in the midplane regions.

The nature of the texture gradients in the second group was very different. This group, distinctive by virtue of strongly depth-dependent textural differences, could be identified with shape factors $\Delta > 1$. An example of these differences, even to the point of a transition to a modified texture in the intermediate regions of the plate is demonstrated in Figure 6 with a sequence of (110)

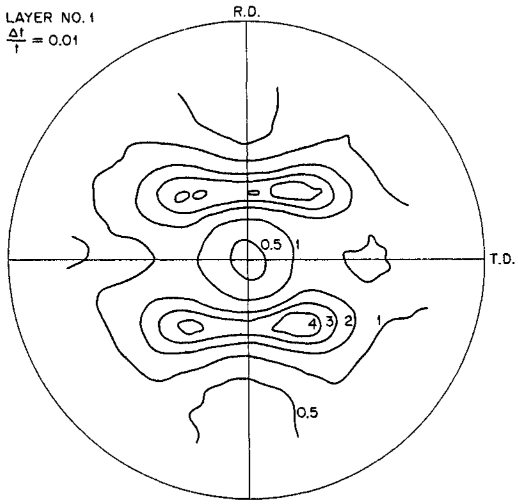

Figure 6a. (110) pole figure of a niobium plate rolled 62% with $\Delta = 2$ at through-the-thickness location $\Delta t/t = 0.01$.

pole figures obtained at various through-the-thickness locations for a niobium plate rolled with $\Delta = 2$. The modified texture which developed at the intermediate depths of $0.17 < \Delta t/t < 0.33$ seemed to contain stable orientations centered on the ideal orientation ($\bar{1}$10)[001] with contributions from material tending to spread toward the ($\bar{1}$10)[112] orientations. Another way the texture inhomogeneity shown in Figure 6 may be displayed graphically is as in Figure 7 where the normalized (110) and

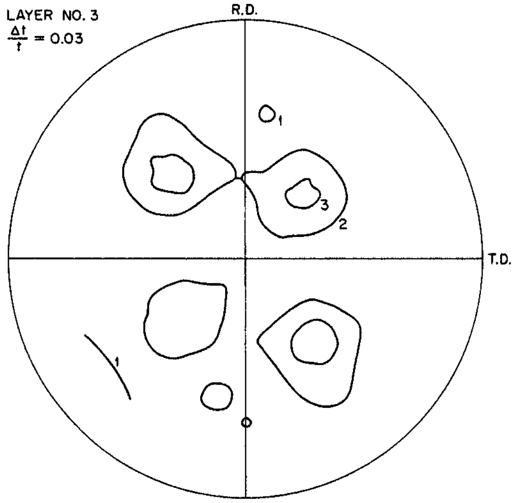

Figure 6b. (110) pole figure of a niobium plate rolled 62% with Δ = 2 at through-the-thickness location Δt/t = 0.03.

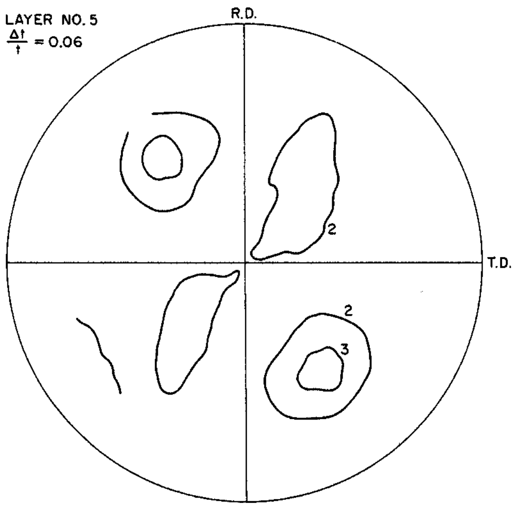

Figure 6c. (110) pole figure of a niobium plate rolled 62% with Δ = 2 at through-the-thickness location Δt/t = 0.06.

(200) intensity in the rolling plane normal direction is plotted versus through-the-thickness location. The same data for Δ = 1/2 are also superimposed on these plots. While the usual rolling texture in niobium exhibits strong (200) intensity and very weak (110) intensity normal to the rolling plane, the modified texture observed in the intermediate layers reversed that pattern.

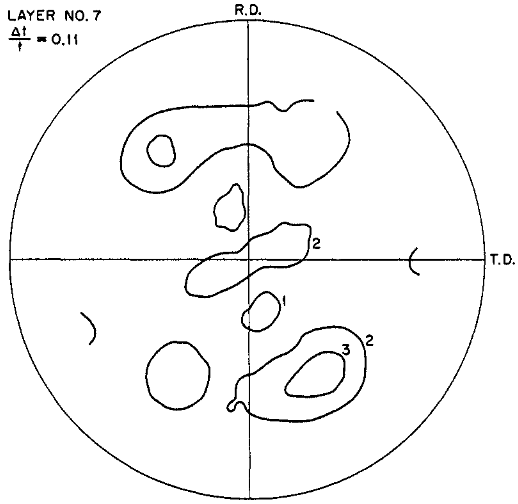

Figure 6d. (110) pole figure of a niobium plate rolled 62% with Δ = 2 at through-the-thickness location Δt/t = 0.11.

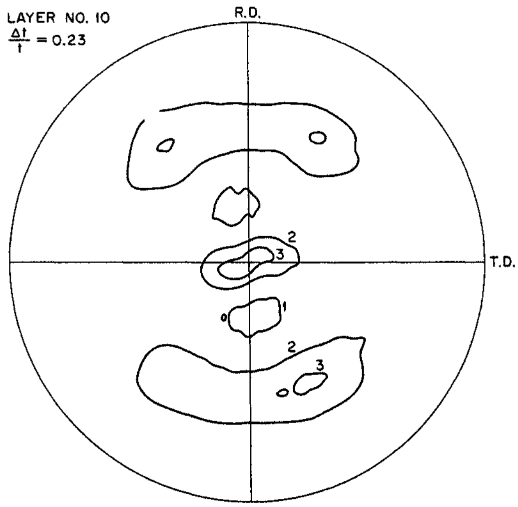

Figure 6e. (110) pole figure of a niobium plate rolled 62% with Δ = 2 at through-the-thickness location Δt/t = 0.23.

In this latter respect, the modified texture resembles the unique surface texture noted for rolled niobium after large amounts of accumulated deformation utilizing heavy drafts and non-lubricated rolls.[5]

At depths immediately above and below the zone of modified texture were transition regions where the pole figures disclosed a smooth and continuous change in

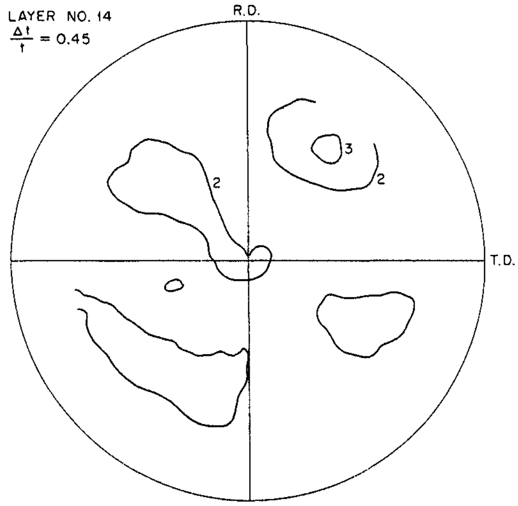

Figure 6f. (110) pole figure of a niobium plate rolled 62% with Δ = 2 at through-the-thickness location Δt/t = 0.45.

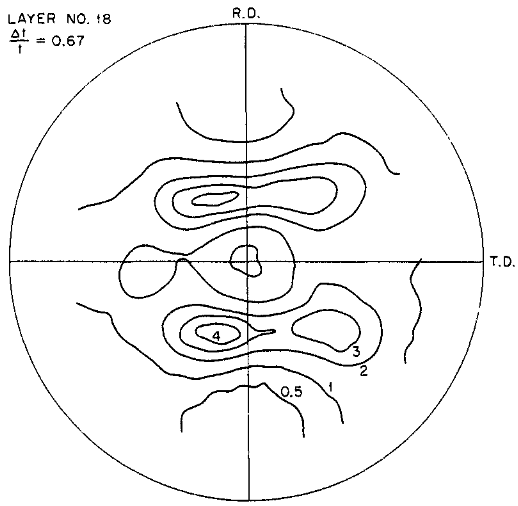

Figure 6g. (110) pole figure of a niobium plate rolled 62% with Δ = 2 at through-the-thickness location Δt/t = 0.67.

orientation with position and no stable orientation was apparent. Particularly noticeable in these transition regions was the lack of four-fold symmetry usually associated with the rolling texture of a bcc metal. Figure 8 attempts to further illustrate this effect and show that the two transition zones tended also to exhibit "anti-symmetric" behavior relative to each other. Curves

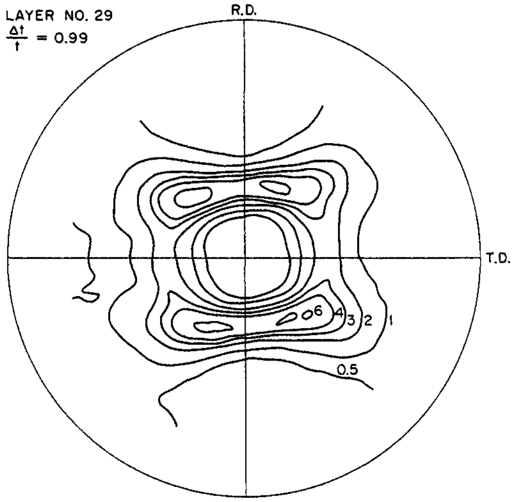

Figure 6h. (110) pole figure of a niobium plate rolled 62% with Δ = 2 at through-the-thickness location Δt/t = 0.99.

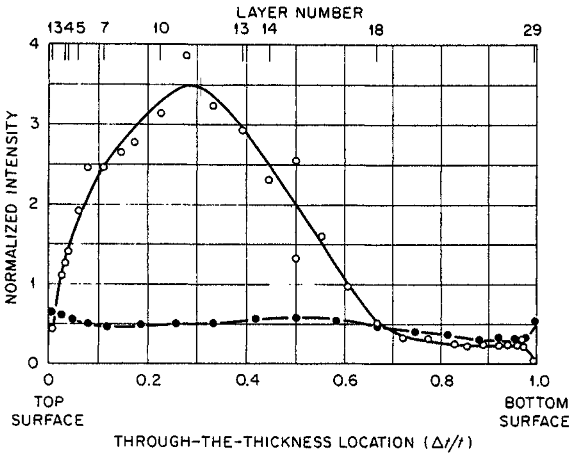

Figure 7a. Depth-dependence of texture in a niobium plate rolled 62% with Δ = 2; normalized (110) intensity in the normal direction. Filled points Δ = 1/2, open points Δ = 2.

A and C represent the depth-dependence of the normalized intensity at pole figure locations corresponding to the supposed ideal rolling $(\bar{1}10)[001]$ orientation in quadrants I and II,* respectively. These intensities should be equal but in the transition zones, they are not, i.e. at $\Delta t/t < 0.17$ and > 0.33. Furthermore, curves A and C cross

*Quadrants I and II of the pole figures are defined in Figure 11.

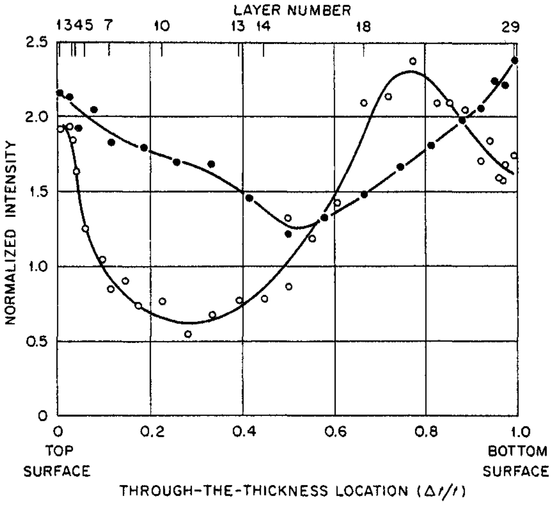

Figure 7b. Depth-dependence of texture in a niobium plate rolled 62% with Δ = 2; normalized (200) intensity in the normal direction.

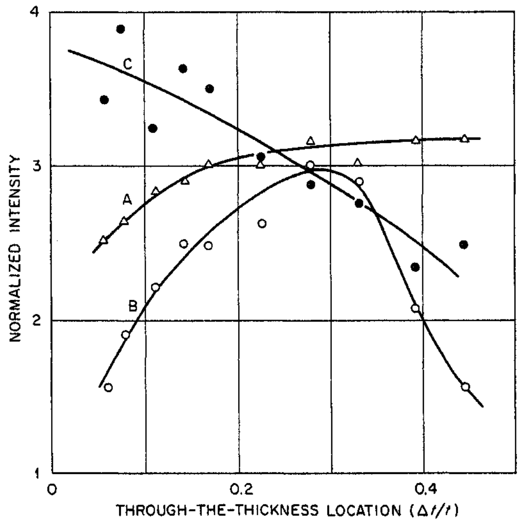

Figure 8. Normalized intensity versus depth in the transition zones. Curve A is the ($\bar{1}10$)[001] intensity in quadrant I; Curve C is the intensity of the same orientation in quadrant II; Curve B is for the ($\bar{1}10$)[112] intensity. (Quadrants are defined in Figure 11.)

over in the region of modified texture suggesting the "anti-symmetric" nature of the two transition zones.

Curve B in Figure 8 represents the depth-dependence of the ($\bar{1}$10)[112] intensity.

In addition to the texture transformation, rolling with shape factors greater than one caused micro-hardness gradients and positional variations in the amount of lateral widening. Figure 9 shows the variation of micro-hardness on a transverse section as a function of thickness for plates rolled with shape factors of 1/2 and 2.

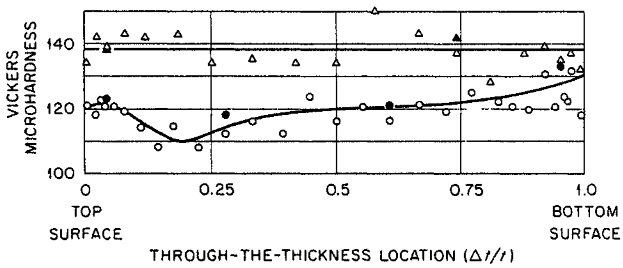

Figure 9. Variation of micro-hardness with through-the-thickness location for niobium plates rolled 62% with Δ = 1/2 (triangles) and Δ = 2 (circles). Filled points represent average of 10 readings.

To a degree, this observation agrees with work by Hundy and Singer.[13] The high Δ rolling (circles) produced a micro-hardness gradient with a minimum corresponding to the location of the deviating texture whereas with low Δ rolling (triangles) the micro-hardness tended to be more uniform. The higher hardness of the low Δ rolled material may be related to the deformation rate, i.e. 4 passes as opposed to 63 to accomplish the same total reduction-in-thickness.

Figure 10 depicts the nature of the laterial widening observed at the edge of a wide plate rolled with shape

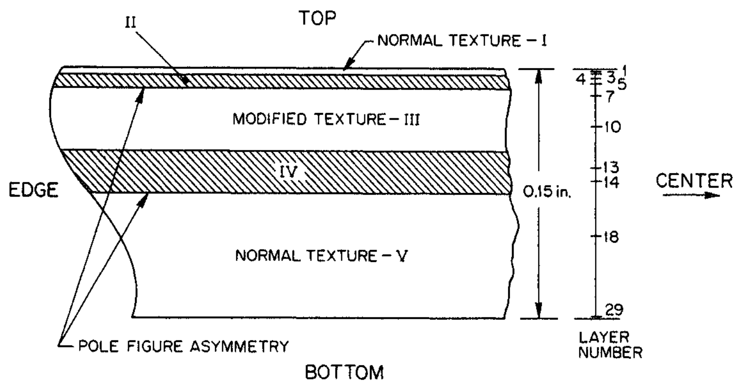

Figure 10. Profile of transverse section through a niobium plate rolled 62% at Δ = 2.

factor of 2. In this profile view of a transverse section, the character of the texture inhomogeneity is superimposed. The maximum degree of lateral widening correlated with the occurrence of the modified texture.

DISCUSSION

It has been demonstrated above that the rolling texture of niobium and particularly through-the-thickness inhomogeneities are strongly influenced by deformation zone geometry as defined by the shape factor Δ. The especially pronounced effects produced by rolling with $\Delta > 1$ are considerably more extreme than the texture gradients detected by Mathur and Backofen[10] in steel for comparable percentage cold reductions. Thus in niobium, new texture components were developed, i.e. the pole figure peak intensities relocated whereas in steel certain pole figure peak intensities changed systematically but the peak locations were not substantially altered.

Past studies have sought to establish relationships between the different textures in a texturally inhomogeneous material in terms of rotations about a common axis parallel to the transverse direction of the work-piece. Figure 11 shows that to be an inadequate description of

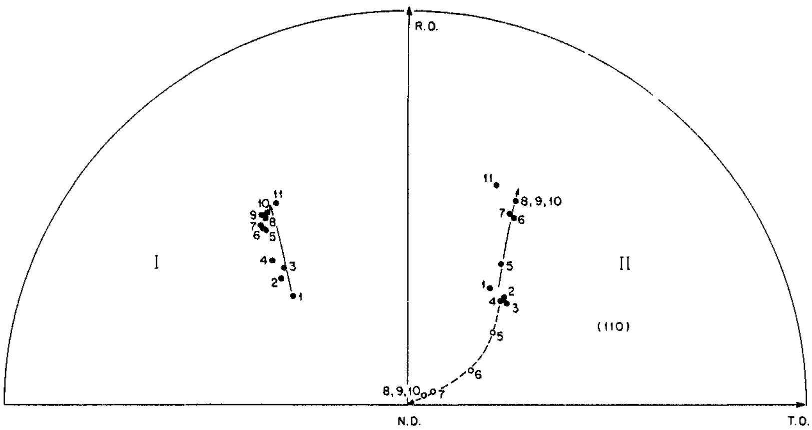

Figure 11. Path of texture relocation in the transition zone of a niobium plate rolled 62% at $\Delta = 2$. Points represent the location of (110) intensity maxima as a function of depth.

the relationship between the modified texture and the normal texture of niobium. In this figure the numbered points plot the location of the (110) intensity maxima in stereographic projection (quadrants I and II only) at various through-the-thickness depths from the top surface (layer 1) with the normal texture through the transition zone, to layer 10 where the modified texture is most pronounced. The solid arrows represent the path of movement of material from an initially ideal (001)[$\bar{1}$10] orientation, a major component of the normal texture, when it is rotated clockwise 25° around the transverse direction. While some of the points (solid) would appear to follow this path, others, i.e. the open points 5, 6, 7, 8, 9, 10 definitely do not. (The fact that some layers are shown twice in quadrant II but only once in I is another manifestation of the lack of fourfold symmetry noted earlier.) Thus it is clear that the transition texture is not related to the normal texture by means of a single, simple rotational relationship such as found by Mathur and Backofen.[10] These results also seem to imply that the modified texture probably develops gradually with increasing deformation from the more-or-less random starting texture rather than as a reorientation from a precursor normal rolling texture. To check this point, several bars of niobium which were rolled with the same Δ but to lower accumulated reduction-in-thickness values were examined. Pole figures obtained from intermediate zones in one bar rolled to only 33% reduction showed evidence already of the transition zone and the modified texture.

The reason why asymmetric pole figures such as Figure 6c for example, were observed in the transition regions between the normal texture and the modified one is not known. Certainly they must imply a very non-homogeneous pattern of deformation in these regions. Additional work is underway to resolve our lack of understanding here.

Roll gap friction has been regarded as being directly responsible for the development of surface textures in Al,[2] Nb,[5] and Fe.[6] These textures, however, do not usually penetrate deeply into the strip even under extreme friction conditions, i.e. no lubricant and large reductions per pass or low Δ. Thus, it would seem extremely doubtful that the modified texture we observed at intermediate depths in plates rolled with Δ = 2, was caused directly by roll gap friction inspite of its similarity to the surface texture produced previously[4,5] under high friction conditions.

The mildly depth-dependent gradients noted for Δ = 1/2, on the other hand, may well be a result of contributions from roll gap friction forces. These forces were apparently not severe enough, however, to promote a new texture. The absence of (112)[$\bar{1}$10] orientations at the surface and the monotonic increase in their intensity in penetrating from the surface to the midplane suggests an alteration in the stress-state of the surface regions due to friction sufficient to cause a lack of stability of this orientation. Without this rationale, it is difficult to account for the missing (112)[$\bar{1}$10] orientations in the surface because they are usually very prominent members of the normal rolling texture of niobium and other bcc materials. In fact the theory of Dillamore and Roberts[14] predicted these orientations to be the most preferred orientation in rolled bcc metals when the stress state is considered to be biaxial with a compressive stress parallel to the rolling plane normal and an equal tensile stress in the longitudinal direction parallel to the rolling direction. With a more complicated stress state that prediction may no longer be valid.

A detailed explanation of the cause of the severe texture gradients for plates rolled with $\Delta > 1$ is not yet possible. It is apparent from these results that here may be another problem area that needs to be added to the list of annoying behavior[15-17] caused by mechanical processing under high Δ conditions.

Tarnovskii *et al.*[1] concluded that the deformation patterns produced by rolling under so-called "high body" conditions, i.e. $\Delta > 4/3$, had many of the attributes of those developed by plane strain compression in the presence of rigid ends. In rolling the rigid ends correspond to the material about to enter the rolls and that having just emerged from the roll gap. With this similarity in mind, Figure 12 is presented showing the Δ dependence

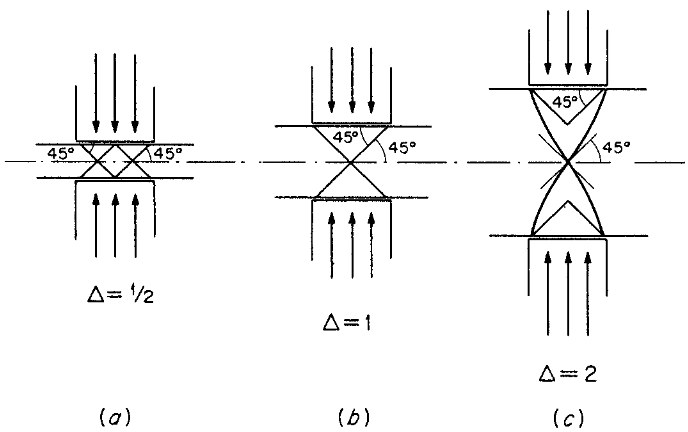

Figure 12. Schematic representation of the Δ-dependence of the slip line fields in frictionless plane strain compression.

of the slip line field, i.e. the family of curves coinciding with the directions of maximum shear, for plane strain compression in the case of a non-strain hardening material deformed between frictionless anvils.

In plane strain compressive deformation, the applied stress state may be regarded as equivalent to a pure shear stress superimposed on a hydrostatic pressure. Backofen[15] pointed out that when $\Delta > 1$ a hydrostatic stress *gradient* would be present, i.e. the hydrostatic stress would vary with position from the surface to the midplane of the work-piece. Furthermore, as Δ becomes increasingly greater than 1, the hydrostatic stress gradient becomes more pronounced and under extreme circumstances[16] the hydrostatic stress itself may even change signs becoming tensile in nature in some regions rather than always being compressive. Coffin and Rogers[17] demonstrated this effect rigorously in a continuum plasticity analysis of plane strain strip drawing of a non-hardening material.

Also it should be emphasized that in frictionless plane strain compression, the maximum shear surfaces, i.e. the slip lines, meet the tool surface and the work-piece midplane at 45° angles and when $\Delta \leq 1$ are straight throughout the thickness (Figure 12, a and b). But for $\Delta > 1$ these surfaces become curved in the intermediate zones (Figure 12c) implying that the principal axes are oriented differently in these regions. It is thought that these "stress-state-gradients" are somehow involved in the generation of severe textural gradients but just how we cannot say.

Finally, a possible rationalization of the way in which the presence of longitudinal curvature can, after many passes, influence the location of the neutral axis, the lack of symmetry about the midplane and the difference in deformation behavior of the top and bottom rolled surfaces is offered in Figure 13. Thus, while the average Δ may have a value of say 2 as in Figure 13a, for a flat plate ,for a given longitudinal curvature the effective (or local) Δ on the upper surface may be larger than the average, perhaps 4, while that on the bottom surface would be smaller, perhaps approaching 1. This is demonstrated in Figure 13b. Volumes near these two surfaces would then be deforming under different apparent deformation zone geometries. The fact that the modified texture and its associated texture gradient is located nearer to the top surface but does not exist near the bottom one, is consistent with the postulate that the higher Δ is above 1, the more inhomogeneous is the deformation. In addition it should be pointed out that the equivalent die angle, α_m, defined in Figure 1, will also be different on the two surfaces. Also, because of the developed curvature and the factors producing it, the lateral widening behavior known as "double-barreling" (Figure 13c) did not result. Based on Tarnovskii's work on the upsetting of cylinders - a process much akin to high body

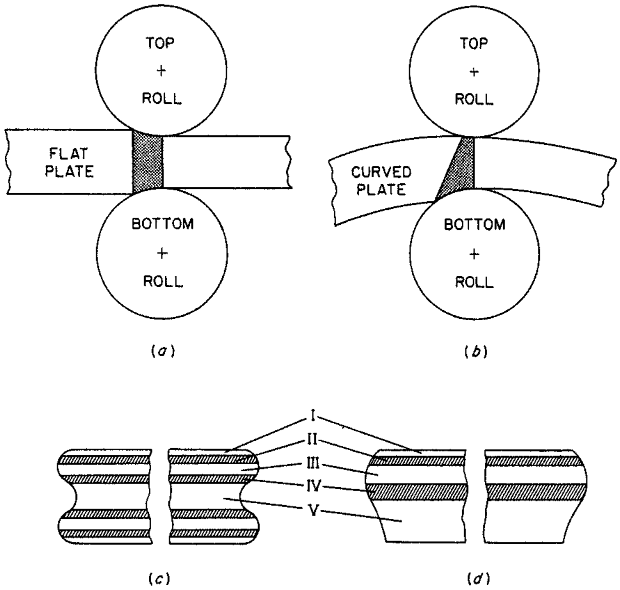

Figure 13. Effect of longitudinal curvature on the deformation zone geometry and lateral widening profile in rolling.

rolling, double barreling was anticipated for rolling with Δ >> 1. Instead, because the top and bottom surfaces apparently deformed differently, a lateral widening asymmetry developed, Figure 13d, - a hybrid so to speak - consistent with the local Δ on the top surface being much greater than 1 thus attempting to form a "double barrel" and that of the bottom surface approaching 1 where no "double barrel" would be expected.

SUMMARY

In this paper it has been demonstrated that the shape (or geometry) of the deformation zone in rolling can importantly affect the formation of texture gradients in cold-rolled plates of niobium.

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