Effect of Annealing Temperature on Plastic Flow Properties and Forming Limit Diagrams of Titanium and Titanium Alloy Sheets

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The influence of annealing temperature on the mechanical properties and deformation behavior of CP-Ti and Ti-4Al-1.5Mn sheets has been investigated. Limit strains in sheet metal have been examined for strain paths between uniaxial and equibiaxial stretching. Uniaxial tensile test has been performed to determine the influence of annealing temperature on the value of strain hardening exponent, n, plastic anisotropy factor, r, and additionally the dependence of the surface roughness growth on the grain size. The relationships between the limit strains and mechanical properties have been examined.

(Received January 18, 1988)

Keywords: forming limit diagram, local necking, grain size, surface roughness, annealing temperature, strain hardening, plastic anisotropy

I. Introduction

The quest for reliable methods of predicting the maximum useful strains, which can be applied to sheet metals in stretch-forming processes, has attracted much practical and theoretical interest in recent years. Our knowledge of behavior in stretch-forming has been advanced significantly by application of Forming Limit Diagrams (FLD)(1). Such diagrams normally refer to stretching performance in proportional straining. They specify the way in which the limiting value of the major surface strain $\varepsilon_1$ varies with the magnitude of the minor surface strain $\varepsilon_2$ in a range of stretching operations, which are characterized by different values of the applied strain ratio, $\rho = \varepsilon_2 / \varepsilon_1$. Sheet materials deforming under multiaxial states of stress, as in sheet metal forming operations, usually fail by localized necking. The current interest in understanding sheet metal formability has led to several theoretical analyses of localized necking based on different criteria. The localized necking criteria include: a localized shear zone along a direction of zero-extention(2), materials imperfections(3), the presence of a vertex on yield surface(4) and voids growth(5).

Available measurements on ductile sheets have shown that the limit strains on the left side of a complete FLD ($\varepsilon_2 < 0$) are usually predicted fairly well by the Hill criterion(1). However, in addition to the flow strength/strain hardening characteristics of the metal, the influence of strain-rate sensitivity may also be significant in some cases. The implication is that, for stretching with zero or negative minor strains $\varepsilon_2$, stretching behavior of ductile sheets could be accounted for in terms of their plastic properties and we need not to be concerned with effects of microstructure except insofar as they influence macroscopic behavior.

The stretchability of sheet metals depends on the materials resistance to localized necking and, in particular, upon material factors which delay the onset of such a plastic instability. The beneficial effects of strain hardening and strain rate hardening on stretchability are well known: both of these effects increase the forming limit strains of sheet metals. It is well known that a high degree of plastic anisotropy as represented by a large $r$-value, which is the ratio of width strain to thickness strain in a uniaxial tensile specimen, promotes formability in drawing. The studies of stretch forming on strongly textured Ti-alloys(6)(7) indicated that a large $r$-value also increase the resistance to localized necking in stretching conditions involving negative minor strains. The effects of

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the r-value on biaxial stretching involving positive minor strains, on the other hands, remain inconclusive because of difficulties in separating the effects of plastic anisotropy from those of strain hardening and strain rate hardening. It has been concluded(8) that the limit strains decrease with increasing the r-value in the $\varepsilon_2 > 0$ region of an FLD but increase with the r-value in the $\varepsilon_2 > 0$ region, and are independent of the r-value at plane strain condition. From the right side of FLD's ($\varepsilon_2 > 0$) there is the evidence that the macroscopic flow strength/strain hardening (and strain rate) relationships are less dominant and the effects of microstructural inhomogeneity are much more important in biaxial stretching(9). Experience has shown that in biaxial stretching, when both the principal strains in the sheet-plane are positive, the strains attained at the onset of localized necking are particularly sensitive to influences of material inhomogeneities. Since the classical theories of tensile instability and necking failure are based on continuum concepts, realistic representations of the effects of plastic inhomogeneities on the scale of microstructure are not easily accommodated.

Almost all the analyses which take explicit account of material inhomogeneities have been based on the method proposed by Marciniak and Kuczyński(3). Originally it was assumed that the inhomogeneity was a geometric defect in a long groove lying perpendicular to the direction of the major principal strain in the sheet plane. However the M-K model can readily be adopted to represent the influence of different kinds of material inhomogeneity provided that the assumption that the weak region is in the form of a long band is retained. Needleman and Triantafyllidis(5) assumed that the weak region is a band of material containing a higher concentration of voids than that in the adjacent areas of the sheet and their analyses of strain localization are based on the constitutive relationship for porous plastic materials proposed by Gurson(10). During deformation voids are created and grow at the particles which cause softening of material. The softening effect of voids increases with the void volume fraction. This means that softening proceeds more rapidly in the particle rich region of the sheet than in the surrounding material, even if the strain levels are identical. Softening promotes strain localization and leads to sharp necking in the particle rich regions of the sheet. Yamaguchi and Mellor(11) published a theoretical model which take explicit account of the influence of grain anisotropy in biaxial stretching. In this it is assumed that up to the stage of stretching at which Swift (diffuse) instability is developed, the amplitude of surface roughening, $R$, will grow in accordance with the relationship proposed by Fukuda et al.(12):

$$ R = R_0 + K \varepsilon $$

where $R_0$ is the initial surface roughness, $K$ a material constant depending on slip characteristics, $d$ the average grain diameter and $\varepsilon$ the effective strain. It was also assumed that incipient grooves are formed within the roughened surfaces and that, in stretching beyond instability, strain localization develops within the deepest grooves in accordance with the M-K model. Melander et al.(13) take account of the model based on the assumption that the necking is initiated by both heterogeneous distribution of particles and surface roughness. Their results have shown that in the cases of titanium sheets the particle contents was so low that a heterogeneity in the particle distribution was not considered a likely cause of necking. For this reason the only heterogeneity existing for titanium sheets was thickness heterogeneity caused by surface roughness.

The purpose of the present paper is the experimental study of:
- the relationships between the limit strains level and Forming Limit Curve (FLC) shape and macroscopic plastic properties,
- the limit strains and plastic properties dependence on microscopic structure (grain size),
- the influence of the grain size on surface roughness growth in tensile test, of titanium sheets annealed at different temperatures.
II. Experimental Procedure

The present investigation was carried out on two titanium sheets—commercially pure titanium sheet CP-Ti and titanium alloy Ti-4Al-1.5Mn sheet. Both sheets have a thickness of 1.0 mm. Tensile specimens and blanks for other tests were cut from the sheets and annealed at 673 K, 723 K, 773 K, 973 K and 1073 K in vacuum (at a pressure less than $2 \times 10^{-3}$ Pa). Since the evident influence of annealing on mechanical properties of titanium sheets appears at the beginning of heat treatment(14), the materials have been annealed for 1.8 ks. The chemical compositions of CP-Ti and Ti-4Al-1.5Mn sheet specimens (before heat treatment) are shown in Table 1.

Tensile specimens, 50 mm in gauge length and 20 mm wide, were prepared from strips cut at $0^\circ$, $45^\circ$ and $90^\circ$ to the rolling direction of the sheet. They will be abbreviated to $0^\circ$, $45^\circ$ and $90^\circ$ specimens hereafter. The experiment has been run using special device which enables to get:

—stress-strain curve and the plot of it in the logarithmic coordinate,
—description of the flow curve with the Hollomon hardening law,
—plastic anisotropy factor, $r$, dependence on the elongation.

Uniaxial tensile testing was performed at room temperature at a constant crosshead speed at an initial strain rate of $4.6 \times 10^{-4} \text{s}^{-1}$.

For the CP-Ti sheet annealed at 673 K the bulge test was carried out. Bulging for equibiaxial stress-strain curve and FLD determination has been run in a hydraulic bulge apparatus using circular die aperture of 100 mm diameter. The bulging pressure has been measured using a strain-gauge transducer and recorded continuously. The curvature of the pole has been determined by a sphereometer device using an axial gauge length of 30 mm.

The stress-strain curves for both tensile and bulge tests were being determined until the maximum load has been reached but the stretching has been continued to the specimen failure to determine the values of surface limit strains in uniaxial and equibiaxial stretching.

The FLDs have been determined basing on the method proposed by Ghosh and Hecker(15).

The average grain diameter has been measured by the linear-intercept method. Grain diameter has been calculated as $d=1.68 l/s$, where $s$ is the number of grain boundry intercepts in a linear transverse of the length $l$.

III. Results and Discussion

The mechanical properties of the CP-Ti and Ti-4Al-1.5Mn sheets are summarized in Table

| Material       | Al | Mn | Mo | V | Composition, mass% |
|----------------|----|----|----|---|--------------------|
|                |    |    |    |   | Cr     | Si   | Fe   | C   | H*    | N   | O   |
| CP-Ti          |    |    |    |   | 0.04   | 0.08 | 0.02 | 0.02 | 0.002 | 0.02 | 0.11|
| Ti-4Al-1.5Mn   | 4.32| 1.33|0.11|0.09| 0.04   | 0.06 | 0.09 | 0.03 | 0.005 | 0.02 | 0.13|

*) Hydrogen content was determined for the all sheets annealed at different temperatures. The influence of the annealing temperature on hydrogen content was not observed.
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Table 2 Mechanical properties and grain sizes of CP-Ti sheets annealed at different temperatures.

| Annealing temperature | Grain diam $\mu$m | Angle to RD | Yield stress MPa | Ultimate streng. MPa | Total elong. % | $n$ | C MPa | $r$ |
|-----------------------|-------------------|-------------|------------------|----------------------|----------------|-----|-------|-----|
| 673 K 8.0             |                  | $0^\circ$   | 375              | 456                  | 29.7           | 0.095| 631   | 2.76|
|                       |                  | $45^\circ$  | 384              | 434                  | 30.7           | 0.071| 573   | 5.11|
|                       |                  | $90^\circ$  | 382              | 448                  | 31.9           | 0.077| 594   | 6.01|
|                       |                  | average     | 382              | 443                  | 30.8           | 0.078| 593   | 4.75|
| 723 K —               |                  | $0^\circ$   | 360              | 441                  | 28.4           | 0.158| 714   | 1.39|
|                       |                  | $45^\circ$  | 370              | 427                  | 29.1           | 0.140| 634   | 2.96|
|                       |                  | $90^\circ$  | 370              | 428                  | 33.1           | 0.146| 677   | 3.12|
|                       |                  | average     | 368              | 431                  | 30.0           | 0.146| 665   | 2.61|
| 773 K 8.9             |                  | $0^\circ$   | 323              | 444                  | 28.4           | 0.173| 706   | 1.62|
|                       |                  | $45^\circ$  | 343              | 414                  | 32.9           | 0.147| 588   | 2.95|
|                       |                  | $90^\circ$  | 354              | 419                  | 30.7           | 0.148| 606   | 3.45|
|                       |                  | average     | 341              | 423                  | 31.2           | 0.154| 622   | 2.74|
| 973 K 12.7            |                  | $0^\circ$   | 258              | 381                  | 34.0           | 0.219| 632   | 2.91|
|                       |                  | $45^\circ$  | 264              | 351                  | 34.7           | 0.193| 599   | 3.19|
|                       |                  | $90^\circ$  | 267              | 363                  | 35.3           | 0.186| 591   | 4.00|
|                       |                  | average     | 263              | 362                  | 34.7           | 0.195| 605   | 3.32|
| 1073 K 17.3           |                  | $0^\circ$   | 230              | 356                  | 37.9           | 0.231| 637   | 3.52|
|                       |                  | $45^\circ$  | 236              | 333                  | 38.7           | 0.197| 552   | 3.95|
|                       |                  | $90^\circ$  | 235              | 326                  | 29.6           | 0.194| 540   | 4.58|
|                       |                  | average     | 234              | 337                  | 36.3           | 0.205| 570   | 4.00|

Table 3 Mechanical properties of Ti-4Al-1.5Mn sheets annealed at different temperatures.

| Annealing temperature | Angle to RD | Yield stress MPa | Ultimate streng. MPa | Total elong. % | $n$ | C MPa | $r$ |
|-----------------------|-------------|------------------|----------------------|----------------|-----|-------|-----|
| 673 K                 | $0^\circ$   | 644              | 763                  | 14.4           | 0.091| 1058  | 1.86|
|                       | $45^\circ$  | 686              | 710                  | 15.2           | 0.062| 984   | 1.93|
|                       | $90^\circ$  | 714              | 750                  | 15.3           | 0.054| 943   | 3.16|
|                       | average     | 685              | 733                  | 15.0           | 0.067| 992   | 2.23|
| 973 K                 | $0^\circ$   | 611              | 754                  | 15.0           | 0.085| 1003  | 1.14|
|                       | $45^\circ$  | 684              | 698                  | 15.5           | 0.079| 914   | 1.49|
|                       | $90^\circ$  | 723              | 733                  | 15.5           | 0.077| 949   | 2.23|
|                       | average     | 675              | 717                  | 15.4           | 0.080| 945   | 1.59|
| 1073 K                | $0^\circ$   | 610              | 746                  | 16.3           | 0.125| 1145  | 0.80|
|                       | $45^\circ$  | 666              | 685                  | 17.0           | 0.083| 905   | 1.66|
|                       | $90^\circ$  | 704              | 714                  | 18.3           | 0.096| 925   | 1.57|
|                       | average     | 664              | 708                  | 17.2           | 0.097| 970   | 1.43|

2 and Table 3 respectively. All properties from the tensile test have been averaged according to:

$$x_{av} = (x_0 + 2x_{45} + x_{90})/4$$

where subscripts refer to the specimens orientation according to the rolling direction.

The results of flow characteristics determination in tensile test showed that the flow curves of the $45^\circ$ and $90^\circ$ specimens differ from the $0^\circ$ specimens (Fig. 1). The maximum tensile load was reached not far after yielding, and the load at failure was 60% to 70% of maximum load. This phenomenon affected the values of strain...
hardening exponent, n, which were visibly smaller for the 45° and 90° specimens than the 0° specimens. It should be noted that the values of total elongation for all the specimens are similar. Large difference in the value of uniform elongation and small difference of total elongation has been caused by the strain concentration in the neck. Distribution of strain components in the vicinity of failure (Fig. 2) has shown that the values and gradients of strain (in longitudinal ε_l and perpendicular ε_w direction of the specimens) of the specimens cut in rolling direction were smaller than those of two other specimens orientation.

The values of the thickness strain ε_t (calculated under the assumption of volume being constant) were several times smaller and their distribution in the uniform and necked region were much more homogeneous. The post-uniform load-carrying capacity (Fig. 1) and the strain distribution in the necked region (Fig. 2) could be explained as a result of crystallographic texture. Titanium alloys in sheet form usually possess crystallographic texture which, in some cases, can be quite strong and often exerts a pronounced influence on the mechanical properties. In the case of sheet metal deformation, a crystallographic texture usually results in plastic anisotropy factor, which could be in the range of r=0.5–12(7). High r-value enhances the post-uniform elongation and the ability to retain the load-carrying capacity beyond maximum load.

The stress-strain characteristics from both tensile and bulge tests have been described using Hollomon’s hardening law. On the basis of the flow curves plotted in the logarithmic coordinate (some examples of these characteristics are presented in Fig. 3) we could conclude that the tensile n-values were constant in the whole range of straining, which caused a good correlation between experimental points and the line corresponding to the Hollomon’s hardening law. In the case of the bulge test the n-value

![Image of flow curves](image1)

**Fig. 1** Flow curves of CP-Ti sheet annealed at 673 K showing the post-uniform load-carrying capacity.

![Image of strain distribution](image2)

**Fig. 2** Distribution of longitudinal ε_l, perpendicular ε_w and thickness ε_t strains in the vicinity of failure of CP–Ti specimens cut at 0°, 45° and 90° according to the rolling direction.
The annealing-temperature dependence of the $r$-value of the CP-Ti sheets seemed to be more complicated (Fig. 5). The large value of plastic anisotropy factor of the sheet annealed at 673 K ($r=4.75$), decreased after annealing at 723 K ($r=2.61$) and then increased with the annealing-temperature in the range of 773–1073 K. Similar relationship was obtained by Lee and Backofen$^{17}$ in the case of Ti-5Al-2.5Sn titanium alloy sheet. Variation of the $r$-value with test direction was very large, especially of CP-Ti sheet annealed at 673 K. The increase of annealing temperature have caused decrease of variation of $r$-value with test direction, both in CP-Ti and Ti-4Al-1.5Mn sheets. The smallest variation of the $r$-value with the annealing temperature has been observed from the test of the $45^\circ$ specimens (Fig. 5).

The onset of localized necking could be explained as a result of thickness inhomogeneity of sheet metal$^{3}$ which is caused by the surface roughness. It was found that the roughness of the CP-Ti sheets from a small initial value increased linearly with the specimens elongation (Fig. 6). The surface roughness was evaluated both as an average value and as an extreme value of the surface profile. The initial value of surface roughness parameters $R_a$ and $R_{tm}$ and the intensity of their growth seemed to depend on specimen orientation to the rolling direction. Variation of roughness parameters $R_a$
and $R_{im}$ with test direction increased with specimens elongation, but became smaller with increasing grain size. The values of roughness parameters of the $45^\circ$ and $90^\circ$ specimens were related. The relationship between the strain value and the values of $R_a$ and $R_{im}$ were linear in the whole range of measured strains (including the necked region in the distance of 5 mm from the failure), but the roughness started to accelerate in the vicinity of failure. The relationships between $R_a$ and $R_{im}$ parameters and effective strain for different grain diameter have been described using the following equations:

$$R_a = Ro_a + S_a \varepsilon$$

$$R_{im} = Ro_{im} + S_i \varepsilon$$

$$R_a = Ro_a + K_a d^{0.5} \varepsilon$$

$$R_{im} = Ro_{im} + k_d d^{0.5} \varepsilon$$

The values of $R_a$, $R_{im}$, $S_a$, $S_i$, $K_a$, $K_i$, $k_a$ and $k_i$ coefficients are summarized in Table 4. The values of initial roughness parameters $Ro_a$ and $Ro_{im}$ for the material tested were related. Variation of the $S$, $K$ and $k$ coefficients with test orientation decreased with increasing grain size. Statistical elaboration (mean value and standard deviation) of $K$ and $k$ values (Table 4) has shown that the eq. (3) give the best fit with the experimental results, better than the eq. (2) elaborated by Fukuda et al.\(^{(12)}\). The value of $K_i = 1.0$ suggests that the roughness growth does not depend on the grain size. The values of $k_i$ coefficients of CP-Ti sheet were related to those of the steel sheets\(^{(18)}\).

The FLD of CP-Ti sheet annealed at 673 K is presented in Fig. 7. The FLC has been drawn below the experimental points of the surface strain components measured in vicinity of fracture or visible necking. During the FLD plotting we have noticed that the experimental points lied below the line of strain ratio $\rho = -0.5$ which was often accepted as a characteristic for uniaxial tensile test. Using the quadratic Hill criterion it was found\(^{(8)}\) that the value of strain ratio, $\rho$, of uniaxial stretching was related to

$$\rho = \varepsilon_2 / \varepsilon_1 = -r / (1 + r).$$

Since the $r$-values of the CP-Ti sheets were much larger than one, the value of strain ratio $\rho = -0.826$ visibly differ from that of isotropic material ($r = -0.5$). The results presented in Fig. 7 indicate that predicted limit strain ratio $\rho = -0.826$ is in good agreement with experimental data. As far as the right region of complete FLD is concerned, the experimental results of limit strains determined in punch-stretching (full circles) and hydraulic bulge (open circles) are in good agreement, although the value of strain ratio of punch-stretching

| Grain diam. $\mu$m | Angle to RD | $R_a^o \mu$m | $S_a \mu$m | $R_{im}^o \mu$m | $S_i \mu$m | $K_a$ | $K_i$ | $k_a$ | $k_i$ |
|---------------------|------------|---------------|------------|----------------|------------|-------|-------|-------|-------|
| 8.0                | 0°         | 0.72          | 2.4        | 4.5            | 11.2       | 0.300 | 1.400 | 0.849 | 3.96  |
|                    | 45°        | 0.67          | 1.4        | 4.2            | 9.2        | 0.175 | 1.152 | 0.495 | 3.25  |
|                    | 90°        | 0.61          | 1.7        | 4.3            | 9.5        | 0.213 | 1.187 | 0.601 | 3.36  |
| 12.7               | 0°         | 0.70          | 2.5        | 5.1            | 12.4       | 0.197 | 0.976 | 0.701 | 3.48  |
|                    | 45°        | 0.65          | 2.0        | 4.1            | 11.8       | 0.157 | 0.933 | 0.561 | 3.31  |
|                    | 90°        | 0.70          | 2.1        | 4.3            | 12.0       | 0.165 | 0.944 | 0.589 | 3.37  |
| 17.3               | 0°         | 0.64          | 3.0        | 4.7            | 15.2       | 0.173 | 0.877 | 0.721 | 3.65  |
|                    | 45°        | 0.65          | 2.7        | 4.6            | 13.5       | 0.156 | 0.788 | 0.649 | 3.25  |
|                    | 90°        | 0.72          | 2.9        | 5.1            | 13.5       | 0.168 | 0.780 | 0.697 | 3.25  |

| Mean value          | 0.189      | 1.000         | 0.651      | 3.43  |
| Standard deviation  | 0.045      | 0.206         | 0.085      | 0.237 |
| (SD/Mean)-100%      | 23.8       | 20.6          | 13.1       | 6.91  |
was \( \rho < 1.0 \).

The FLCs for all of the materials tested are summarized in Fig. 8. The level of limit strains of the Ti-4Al-1.5Mn sheets is visibly smaller than that of CP-Ti sheets. The FLCs of both CP-Ti and Ti-4Al-1.5Mn sheets are characterized by a large negative minor strain regime \( (\varepsilon_2 < 0) \) but relatively smaller positive minor strain region \( (\varepsilon_2 > 0) \). Annealing of the sheets in the temperature range of 773-1073 K enhanced the level of limit strains of CP-Ti sheet in higher degree than that of Ti-4Al-1.5Mn sheet. The FLCs of the sheet annealed at high temperatures became more flat, especially in the range of \( \varepsilon_2 > 0 \). Hill's theory predicts that the maximum principal strain \( \varepsilon_1 \) prior to localized necking (i.e. the limit strain) has a magnitude of \( \varepsilon_1^p = n \) at plane strain and increases to \( \varepsilon_1^p = (1 + r)n \) for uniaxial region. A good agreement of experimental points with prediction of Hill's theory for the uniaxial tension has been observed only in Ti-4Al-1.5Mn sheets and CP-Ti sheets annealed at 673 K and 773 K. The magnitude of limit strains at the plane strain for all the materials tested was larger than that predicted according to Hill's theory. The value of limit strains at plane strain were found to be \( \varepsilon_1^p = 1.34n \) in Ti-4Al-1.5Mn sheets and to be \( \varepsilon_1^p = 0.09 + 1.3n \) in CP-Ti sheets (except of the sheet annealed at 1073 K). The value of limit strains of CP-Ti sheet annealed at 1073K characterized by the largest n-value, were smaller in the whole range of the FLD than that of CP-Ti sheet annealed at 973 K. The CP-Ti sheet annealed at 1073 K was characterized by the largest grain size and thus the surface roughness growth during stretching was the most intensive, which caused the decrease of limit strains. In the biaxial stretching region of the FLD the effect of macroscopic plastic properties, like the \( n \)-value and the \( r \)-value, on the level of limit strains cannot be directly established. The increase of \( n \)-value and decrease of \( r \)-value accompanied with the increase of annealing temperature of the Ti-4Al-1.5Mn sheet should have caused more intensive increase of the limit strains in biaxial stretching. In the case of the CP-Ti sheets no universal relationships were found. Thus the effect of microstructural inhomogeneity on the level of FLC in biaxial stretching seemed to be much more important than that of macroscopic plastic behavior of the sheet.

**IV. Conclusions**

1. Annealing in the temperature range of 673 K to 1073 K strongly affects the mechanical behavior of CP-Ti sheet. In the case of Ti-4Al-1.5Mn sheet the influence of annealing temperature on the mechanical behavior was visibly smaller. The value of strain hardening exponent, \( n \), increases with increasing annealing temperature. The value of plastic anisotropy factor, \( r \), of Ti-4Al-1.5Mn sheet decreases with increasing annealing temperature, but that of CP-Ti sheet varies in a complicated manner with annealing tempera-
tecture. In both cases the variation in $r$-value with the test direction decreases with increasing annealing temperature.

(2) The values of limit strains in uniaxial tension could be predicted using Hill's criterion ($\varepsilon^i = (1 + r)n$) for almost all the materials tested (excluding the CP-Ti sheets annealed at 973 K and 1073 K). The values of limit strains at plane strain were larger than those predicted using Hill's criterion ($\varepsilon^p = n$). There was no apparent relationship between limit strains in biaxial stretching and macroscopic plastic flow properties of the materials—the effects of microstructural inhomogeneity are dominant.

(3) The value of limit strains depends on geometric inhomogeneity. The growth of the surface roughness of sheet metal in stretching process can be well represented as a linear function of effective strain and square root of the average grain diameter.

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