Relation between Deformability and Microstructures in a Commercial Pure Ti Sheet Subjected to Dual-temperature Square-shaped Deep Drawing

Jaan-Ming LIU, In-Gann CHEN1) and Sheh-Shon CHOU

Graduate student, Department of Materials Science and Engineering, National Cheng Kung University, Tainan, Taiwan, 70101, Republic of China. 1) Department of Materials Science and Engineering, National Cheng Kung University, Tainan, Taiwan, 70101, Republic of China. E-mail: ingann@mail.ncku.edu.tw

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Recent studies indicated that the deep drawing process at dual-temperatures could extend the drawability in many materials. This paper reports the study of commercial pure Titanium (CP-Ti)(α-Ti) heated to the temperature range of 25–400°C and deep-drawn by water-cooled square-shaped punch. The CP-Ti exhibits almost triple drawability when the temperature is increased from room temperature to 400°C. The location of the fracture point of CP-Ti sheets is on the corner of the cup wall near the die throat. The poor drawability at low temperature is due to the strain-hardening caused by reorientation bands (RBs). The active slip system at elevated temperature promotes the drawability of CP-Ti. The X-ray diffraction studies show the tendency of slip system to be active on the prismatic plane at −200°C and on the pyramidal plane at −400°C. The process at 400°C shows the microstructure with the micro-shear bands (MSBs) intersection induced by the heated die and the cooled punch. The MSB-MSB intersection prevents the further thinning of the cup wall and results in the superior deep drawability by virtue of better ductility.

KEY WORDS: commercial pure Titanium; microstructure; dual-temperature deep drawing; shear band; slip.

1. Introduction

One of the deep drawing properties is the limiting drawing ratio (LDR), which can be improved by suitable combination of the processing parameters and the material characteristics. The local instability1) on the drawn cup wall due to the interaction between the punch force and the blank holding force (BHF) generally lead to fracture. The materials characteristics, such as the plastic anisotropy value R2) and work hardening coefficient n,3) which dominate LDR have been discussed in various studies.4–7) The operating temperatures also play an important role on the drawability while sheet metal was processed by square-shaped deep drawing. The processes utilizing dual-temperatures, i.e., heated blank holder and water-cooled punch, which can provide a better drawability than the normal single temperature condition for many metals, such as, stainless steel,8,9) aluminum alloys,10,11) and titanium alloys.12–16)

The slip system and deformation twinning are two major plastic deformation mechanisms, which have been widely investigated in α-titanium of hcp structure.17–19) Here are more than one slip systems in α-Ti, which include basal, prismatic, and pyramidal slip systems with \( \{1120\} \) Burgers’ vector, and the pyramidal slip system with \( \{\overline{1}3\overline{1}2\\} \) Burgers’ vector.17) The twinning systems are \( \{110\} \) (1101), \( \{12\overline{1}2\} \) (1213), and \( \{1\overline{2}1\} \) (1216) twin.17,19) The plastic deformation of single crystalline titanium at elevated temperature had also been explored.18) Previous studies of the deep drawability of sheet metals were emphasized on the mechanical properties. The present work explores the detail of the deformation microstructure induced by the dual-temperature deep drawing process and its correlation with the drawability of commercial pure titanium (CP-Ti).

2. Experimental Procedures

The CP-Ti sheets (AMS 4901 grade 4) were used in this study with the chemical composition (in mass wt%) of 0.01% C, 0.12% Fe, 0.34% O, 0.011% N, 0.026% H, and the balance Ti. The deep drawing press is an AMINO universal forming test machine similar to a previous study on CP-Ti sheet,15,16) and the die alignment is shown in Fig. 1. The octahedral-shaped sheet specimens, 140 mm width and 0.88 mm thick, were subjected to deep drawing at temperature 25°C, 100°C, 200°C, 300°C and 400°C with 50 mm width square-shaped punch. The blank holder, of 8 mm-radius shoulder and TiC coating on the both surfaces, was preheated to the selected temperature. The flat-end punch was cooled by pumping ice water through the drilled center axis. Before pressing, the specimen was cut into the selected size and a grid etched upon it for the purpose of positioning. When the mold was heated to the operating temperature, MoS2 was applied to the surface of the specimen,
and the specimen was placed between the blank holders for at least 3 min to heat to the selected temperature. Then the specimen was forced to flow into the mold cavity by the water-cooled punch with 4 mm/sec drawing speed. The blank holding force (BHF) was applied by controlling the displacement of the disc-springs. The TEM specimens were cut off from the cup wall near the fracture region. X-ray and TEM analyses were used to explore the deformed microstructure and its correlationship with the variation of formability limit at different operating conditions.

3. Results and Discussions

The appearances of the drawn cups with different deep drawing conditions are shown in Fig. 2.

3.1. Operating Temperature and Blank Holding Force

Illustrations in Fig. 3 are the drawing heights of the drawn cups and the BHF loading in different operating temperatures. The deep drawability increases from room temperature to 400°C. The drawing height of the drawn cup at 400°C is about three times larger than that at 25°C. The material on the flange portion deforms more easily at elevated temperature, which is believed to be due to the relative softness of the material strength. And the material on the flange portion can be pulled to flow into the mold cavity more easily, which is believed to be due to the strengthening of the material on the cup wall by the cooling effect from the punch. Therefore, the formability at elevated temperature becomes better as work hardening effect becomes milder than that at room temperature. A drop on of the drawing height was observed at 300°C can be attributed to the low thermal conductivity (k) of the material (k = 17.1, 16.9, 16.7, 17.2 W/m·K, at 100, 200, 300, 400°C respectively), where the cooling effect from the punch is insufficient to promote the strengthening of the cup wall.

Under different operation temperatures and holding forces, almost all the fracture occur at the similar position which is on the corner of the cup wall near the flange portion. It is noted that the failure point of CP-Ti (hexagonal crystal system) is different from that of the stainless steel and aluminum alloys (cubic crystal systems). The failure point of CP-Ti is on the cup wall corner near the die throat (marked as B in Fig. 4), which is probably due to plastic anisotropy, and those of the stainless steel and alu-

![Fig. 1. Die alignment of the deep drawing equipment.](image)

![Fig. 2. Appearance of the drawn cups. The "**" represents the formability limit in the selected operating temperature. The number under each picture is the blank holding force (kgw).](image)
minimum alloys are reported primarily at the edge of the cup bottom\(^{8-11}\) (marked as A in Fig. 4). From Fig. 2 (f) and (g), it is observed that the fractures spread out along the rolling direction (R.D.). It is believed that a non-uniformly tension occurs on the cup wall of CP-Ti, which causes necking by thinning on the easy tension direction normal to R.D. and initiates the fracture.

As shown in Fig. 2(e) and 2(k), the low BHF causes wrinkles around the specimen on the flange portion, and this wrinkle will extend to the cup wall. No wrinkle are shown in Fig. 2 (a)–(c), (f), (g), and (l), where the high BHF restricts the metal plastic flow. The non-uniform residual stress in the flange portion of the specimen causes bending after the BHF was released at room temperature. It is noted that a higher BHF is required as the operating temperature increases up to 400°C. In order to avoid wrinkle and reach the full drawing height a larger BHF is required.

3.2. Thickness Strain Distribution of the Drawn Cup

Figure 4 shows the effect of different operating temperatures on the distribution of the thickness strain, i.e., the degree of thinning, of the drawn cups along the direction of 45° to R.D., i.e. the direction across the corner of the drawn cups. In the insert of Fig. 4, the mark A and B represent the point of the cup wall near the cup bottom and that near the flange portion respectively. In point A, the degree of thinning decreases from room temperature to elevated temperature. It reveals that at elevated temperature the cooling effect from the punch enhances the strength of point A and allows the further thinning without fracture. In point B, where CP-Ti is thinned and the fracture occurs due to the initiation of local necking. This effect is different from that of the cubic structure metals, such as Al and Fe, where the fracture is usually caused by the thinning and plastic instability at the corner (A). It indicated that the CP-Ti material of the flange portion (B) exhibits more ductility than the portion of corner (A) at elevated temperature, and CP-Ti is thinned more significantly at B to form the cup wall. By comparing the thickness strain of the point A and B for samples operated at 25°C and 100°C, the thinning phenomena of these two points are similar. For temperatures above 200°C, the thickness strain of the point B is greater than that in point A. This indicates that the CP-Ti material on the cup wall is thinned gradually and homogeneously above 200°C due to the better ductility caused by the effective temperature gradient on the cup wall. It is noteworthy that in the cup wall of sample operated at 400°C, a uniform region of the thickness strain, marked as C, which extends over about 20 mm distance can be observed. In other words, the local necking does not occur in the extended C region and the best formability of CP-Ti is achieved.

3.3. Preferred Orientation Influence on Deformation

Figure 5 shows the variation of texture coefficient ($T_{\text{chki}}$) as a function of operation temperatures and crystallographic orientations. The texture coefficient is calculated from X-ray diffraction data based on the definition\(^3,24\):

$$T_{\text{chki}} = \frac{(I_{\text{hkl}}/I_0)}{(1/n) \sum (I_{\text{hkl}}/I_0)} \quad (1)$$

where $I_0$: the integral intensity before the deformation

$I_{\text{hkl}}$: the integral intensity after the deformation of
the crystal orientation ($hkil$: the Miller indices of the plane)

$n$: the number of reflections

There is no unusual variation in $TC_{hkil}$ in the operations at 25°C and 100°C. The low magnitude of $TC_{hkil}$ variation in different crystal orientations is attributed to the low plastic deformation and the lack of slip activity in a particular slip system. At 200°C operation temperature, $TC_{11\bar{2}0}$ increases to $\sim 1.52$ (vs. $\sim 1.06$ at 100°C) and $TC_{01\bar{1}2}$ reduces to $\sim 0.49$ (vs. $\sim 1.01$ at 100°C). It reveals that the slips on the (11\bar{2}0) prismatic plane glide more easily than those on the (01\bar{1}2) pyramidal plane. However, at 400°C operation temperature, $TC_{11\bar{2}0}$ reduced to $\sim 0.58$ and $TC_{01\bar{1}2}$ increases to $\sim 1.03$. This reverse in the tendency of slip system in 400°C operation temperature indicates that the active slip system changes from the (11\bar{2}0) prismatic plane to the (01\bar{1}2) pyramidal plane. Therefore, the enhancement in drawability at 400°C operating temperature can be attributed to the different active slip system induced.

### 3.4. Failure Point Microstructure Analysis

The microstructures of the failure points in different operating temperatures were observed by transmission electron microscope (TEM) to investigate the deformation mode during the dual-temperature deep drawing process. At the initial stage of drawing, i.e., before the cup wall was deep-drawn, a complex effective stress, which includes the tensile radial stress, compressive hoop stress, and blank holding force, is applied to the material on the flange portion. The stress condition varies, as the material is deep-drawn from the flange portion into the die-throat. In this situation, the material is just flowing into the mold cavity from the flange portion and the temperature becomes slightly lower than the heated flange region by the water-cooled punch. The material is lengthened in the radial direction and compressed in the hoop direction gradually. It is observed that the most likely failure point is on the corner of the cup wall near the die throat marked as “B” in Fig. 4, where only the tensile radial stress is applied to lengthen material in the radial direction and the thickness is reduced gradually.

The TEM micrographs of CP-Ti sample before deep drawing process are shown elsewhere.$^{15,16}$ Only the parallel straight $\bar{a}$-dislocation clusters in two directions and the dislocation tangles around these $\bar{a}$-dislocations are observed.

### 3.4.1. Microstructure in Low Operating Temperature

For the sample deep drawn at room temperature, the microstructure with reorientation bands (RBs)$^{25}$ is observed. In other words, the grains are re-oriented during the deformation and propagate into elongated grain with RBs under the applied stress as shown in the TEM micrograph of Fig. 6(a). Figure 6(b) is the dark field image of Fig. 6(a) in different magnification to reveal the crystallographic orientation relationship. In the insert of Fig. 6(a), it is noticed that a distinct direction relative to its neighbor grain dominates the deformation mode. Listed in Table 1 is the rotation angle of the TEM foil and the identified zone axis of the selected area in the insert of Fig. 6(a).

| Selected Area | Angle of Rotation | Identified Zone Axis |
|---------------|------------------|----------------------|
| (1)           | 18.2             | [2 1 10]             |
| (2)           | 29.2             | [2 1 10]             |
| (3)           | 15.4             | [2 1 10]             |

The dislocation density is low and no deformation microstructures can be observed.

Fig. 5. Texture coefficient at different operating temperatures and crystallographic orientations.

![Fig. 5. Texture coefficient at different operating temperatures and crystallographic orientations.](image)

![Fig. 6. TEM photograph of sample deformed at room temperature (i.e. sample (c) of Fig. 2). (a) Bright field image of reorientation bands propagation and (b) its dark field image on (0001) reflection.](image)

![Table 1. The rotation angle of and the identified zone axis of the Selected Area in the insert of Fig. 6(a).](image)
lected area in the insert of Fig. 6(a), indicating that the orientations in these neighbor grains are similar with low angle boundaries among the grains. The RB acts as a hardening region and accelerator of storage of dislocation. This region also behaves as an obstacle for slip and results in dislocation density increase in the grain. Since the RBs are induced by the increasing of plastic strain, the accumulation of dislocations results in work hardening of CP-Ti. An optimum rate of RBs formation and work hardening is required for the maximization of formability in CP-Ti deep drawing process. The rate of formation must be high enough to prevent the onset of necking, but must not exceed the fracture limit of the fragile region. The continuous increase in plastic deformation creates extensive RBs and increase in both the flow stress and the strain hardening rate. Finally, the fracture occurs when the RBs formation becomes saturated and a rapid decrease of the work hardening rate yields a premature local plastic instability. The drawability remains poor due to the low ductility at 100°C.

3.4.2. Microstructure in 200–300°C Operating Temperature

The extent of working hardening decrease as the operating temperature increases. The deformation stress can hardly initiate the RBs formation at elevated temperature and the plastic deformation occurs entirely by slip system. For the sample deep drawn at 200°C, Fig. 8(a) is a TEM micrograph which shows a homogeneous glide in a single micro-shear bands (MSBs). And Fig. 8(b) is its dark field image to reveal this single MSBs. The RBs propagation and its dark field image are shown in Figs. 8(c) and 8(d) respectively. As illustrated in Figs. 8(c) and 8(d), the RBs with a larger width (compared with that in Fig. 7) represents a decreased work hardening rate, and the RBs may propagate into MSBs within a short period of time. The observation is consistent with the XRD texture coefficient measurement in Fig. 5 which shows a texture orientation in (11¯20) plane for sample with 200°C operating temperature. Because the grain is weakened by increasing temperature, the external stress is strong enough to cause the MSB plastic deformation. Hence, the shear deformation occurs homogeneously along the direction of the applied stress, which leads to failure. The interaction of single MSBs and the relatively weak RBs increase the drawability of CP-Ti operated at 200°C slightly.

When the operating temperature is increased to 300°C, the drawability becomes even worse than that of 200°C. Figure 9(a) is the TEM micrograph which shows the individual MSBs aligned together in the form of MSB bundles in the same direction and Fig. 9(b) is its dark field image. It is noted that the slip system on samples operating at 300°C is different from that of 200°C. It is reported that the thermal conductivity of Ti decreased from 16.9 (W/m · K) at 200°C to 16.7 at 300°C and then increased to 17.2 at 400°C. This decrease in thermal conductivity at 300°C could result in a slower heat flow between the water-cooled punch and the heated Ti sheet than that of the other operating temperature. Therefore, the Ti sheet deep drawn at 300°C will experience a different temperature profile and slip system. As shown in the following section (Sec. 4.3) for samples operated at 400°C, there are two MSBs interacting with each other to relax the internal stress accumulation. For samples operated at 300°C, there is only one active MSBs observed. A similar MSBs is also observed for sample operated at 400°C to be induced at high temperature and low flow stress. It is believed that the low thermal conductivity at 300°C slow down the cooling process while the water-cooled punch was applied. The secondary MSBs as shown in Fig. 10 for samples operated at 400°C, which is
believed been induced at low temperature with a higher flow stress does not exist. The accumulation of dislocations only in the MSB can nucleate the cleavage fracture.\textsuperscript{30,31}) Once a crack nucleation occurs, the growth of crack is very rapid and unable to stop. The less drawability of CP-Ti at 300°C than 200°C is caused by the premature fracture at the cup-wall, though the material on the flange is more ductile.

3.4.3. Microstructure in 400°C Operating Performance

It is known that the internal stresses due to the accumulated dislocations or the interaction of dislocation forest can be relaxed by the slipping on the secondary systems\textsuperscript{32,33}) Since the punch was water-cooled and the die was heated to 400°C, a larger temperature difference exist. As shown in Fig. 5, the texture coefficient of \((11\bar{2}0)\) and \((11\bar{1}2)\) plane are significantly varied in the temperature range of 200–400°C. These results in Fig. 5 suggest that two different slip systems will be induced at different portion of the Ti sheet due to this temperature difference. Varied straining due to the temperature difference on either side of the Ti sheet caused slip on the different MSBs, and these two types of MSBs interacted with each other to form stable dislocation tangles that relaxed internal stresses continually during the deep-drawing process.

In order to fully elucidate the progress of the deformed microstructure change when the Ti sheet was plastically flow from the heated flange region to the die throat region, two TEM samples were cut separately from two Ti sheets deep drawn to 30 mm and 50 mm height respectively at 400°C operating temperature. These two samples with positions shown as small circles in the insert of Fig. 10(a) were selected for TEM analysis. The MSB-MSB intersection appeared at the region on the cup wall near the die throat with the half drawing height of about 30 mm as shown in Fig. 10. The MSB-MSB intersection and its microstructure in TEM micrograph are shown in Figs. 10(a) and 10(b) respectively. The TEM micrographs, which reveal these two different MSBs, and their dark field images are shown in Figs. 10(c)–10(f) respectively. It is known that the flow stress, yield stress, and yield strain of CP-Ti decrease significantly with increasing temperature. The different types of stress–strain conditions caused by the different temperatures on both sides of Ti sheet will generate more than one MSB and impinge each other. There are more active slip systems when deformed at elevated temperature than at low temper-
Fig. 9. TEM photograph of sample deformed at 300°C (i.e. sample (k) of Fig. 2). (a) Bright field image of MSB bundles and (b) its dark field image on (1010) reflection.

Fig. 10. TEM photograph of sample deformed at 400°C with the half drawing height (i.e. sample (m) of Fig. 2). (a) Bright field image of MSB intersection with 8000× magnification and (b) microstructure in the MSB intersection region with 50000× magnification. (c) Bright field image of one MSB and (d) its dark field image on (1210) reflection. (e) Bright field image of the other MSB and (f) its dark field image on (1100) reflection.
ature, and the amount of slipping on each MSB system is more uniformly distributed at elevated temperature. Therefore, the slip lines or bands of the first formed MSB act as obstacles to the secondary one and interact with each other to relieve stress concentration. The shear offsets created at intersections also exhaust some strain energy and transform into the shear strain. Then the grains were work-hardened to relax the strain energy and strong enough to resist shear instability.

A greater difference in temperatures between the two sides generates a larger gradient of flow stress in grains as the temperature is increased to 400°C, and accumulated stresses are relieved by MSB-MSB intersections. Accumulation of dislocations on the first slip plane (probably at the elevated temperature side due to a lower flow stress) can act as slipping sources on one side when the secondary slip dislocations (probably at the lower temperature side due to a higher flow stress) intersect them. Similar accumulation of dislocations produced by the secondary slip dislocations is sources of slipping on the other side. Thus, the MSB-MSB intersections are formed and occur on both planes throughout the crystal grain and the internal stress is the same on both MSBs. The MSB–MSB intersections result in a remarkable reduction in the width (marked as W) of MSB ~0.1–0.15 μm (vs. ~0.26 μm in Fig. 8 of 200°C and ~0.5 μm in Fig. 9 of 300°C) and store less internal energy. Therefore, the slips are more stable.

When the cup is drawn to the full drawing height of 50 mm, long MSB clusters without secondary MSB are observed in Fig. 11. The MSB clusters and its dark field image are shown in Figs. 11(a) and 11(b) respectively. In the insert of Fig. 10(a), the positions of TEM samples illustrate the relationship of Fig. 10 and Fig. 11, i.e., the middle and the final stage of deep drawing. In Fig. 11(a), the spacing (marked as S) between MSB clusters is ~0.5 μm, and in Fig. 10(a), the spacing is ~3.5 μm. The smaller spacing in the final stage of deep drawing process indicates that the primary MSBs induced on the flange portion were stacking closer as the plastic deformation proceeds. It is believed that the long distance glide in the MBS clusters can relax the internal stress of the lengthened grain due to the softening of the material on the flange portion. The secondary MSBs induced by the cooled punch as shown in Fig. 10 was not observed. The single MSBs in the final stage of deep drawing can be rationalized as follows. As the thickness of Ti sheet decreased in the middle stage of drawing, the contact pressure between the cooled punch and the Ti sheet reduced. The cooling effect that result in the temperature gradient between two sides of the Ti sheet also decreased. In addition, the stress configuration in region C changes to the plane stress, not the thickness stress, due to a uniform thickness strain. Therefore, only one MSBs was induced in this final stage of deep drawing.

This series of TEM analyses on samples operated at different temperatures and positions provides a detail understanding on the reason that the cup wall forms a uniform region of thickness strain, which extends over 20 mm distance marked as C in Fig 4. And the CP-Ti exhibits the best formability at 400°C in the square shell deep drawing process.

4. Conclusions

Typically, the square-shaped deep drawing at dual-temperature can effectively enhance formability of commercial pure Titanium sheets at 200°C and 400°C, but worse at 300°C. The fracture point of CP-Ti cup is on the corner of the cup wall near the die throat, not near the punch shoulder as that of cubic crystalline metals. For the CP-Ti cup drawn at low temperatures (<100°C), the strongly strain-hardening effect due to the reorientation bands causes the low drawability. As the tendency of slip system occurs more easily on the prismatic plane at 200°C and on the pyramidal plane at 400°C, the active micro-shear bands (MSBs) promote the drawability. The MSB–MSB intersections observed on specimen deep drawn at 400°C with the primary MSBs induced by the heated die and the secondary MSBs induced by the cooled punch. It is believed that this MSB intersection prevents the local plastic instability and allows further thinning on the cup wall to cause almost triple drawability compared with that at room temperature. Hence the best formability of the squared-shaped deep drawn CP-Ti cup is achieved at 400°C.
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