# MECHANICAL BEHAVIOR OF COPPER THIN FILMS SUBJECTED TO VARIOUS STRAIN RATE LOADINGS

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## ABSTRACT

In this study, a micro-force tensile testing machine (MTS Tytron 250) was applied to test the polyimide samples coated with different thicknesses of copper (500–1500 nm). The experiments using different strain rates ($1.6\times10^{-4}$ to $1.6\times10^{-2}$ s$^{-1}$) were conducted to the test vehicles. The results showed that the stress and strain of Cu films were strongly correlated with the strain rate and film thickness. The mechanical strength, yield stress, Young's modulus, and maximum tensile stress, increase as the strain rate increases or the thickness decreases. Strain rate sensitivity rapidly increases as the thickness decreases from 750 to 500 nm to imply that the workhardening rate increases while the thickness decreases, resulting in a higher probability of brittle failure.

**Keywords:** copper thin film; strain rate sensitivity; mechanical behavior; tensile test.

# COMPORTEMENT MÉCANIQUE DE COUCHES MINCES DE CUIVRE SOUMISES À DES VITESSES VARIÉES DE DÉFORMATION

## RÉSUMÉ

Dans cette étude, le système d'essai de matériaux pour des tests de "micro-force" (MTS Tytron 250) a été appliqué pour tester des échantillons de polyimide enduit de différentes épaisseurs de cuivre (500–1500 nm). Des expériences avec différentes vitesses de déformation ($1.6\times10^{-4}$ to $1.6\times10^{-2}$ s$^{-1}$) sont menées sur le véhicule utilisé. Les résultats montrent que le stress et la déformation de couches de Cu étaient fortement en corrélation avec la vitesse de déformation et l'épaisseur de la couche. La force mécanique, l'effort de tension, module de Young, et la contrainte maximum de tension s'accroît en augmentant la vitesse ou en diminuant l'épaisseur. La sensibilité augmente rapidement en diminuant l'épaisseur de 750 à 500 nm, laissant supposer que la vitesse de rigidité augmente alors que l'épaisseur diminue menant à des probabilités plus élevées de rupture cassante.

**Mots-clés :** couches minces de cuivre ; vitesse de déformation ; comportement mécanique ; essai de tension.

## 1. INTRODUCTION

The sizes of electronic devices, in particular, cell phones, computers, MEMS, detectors are toward small and small due to rapid development of semiconductor process in order to maintain the Moore's law, stated by a rule of the number of transistors that can be placed inexpensively on an integrated circuit doubles approximately every two years. The thin film materials are often subjected to mechanical-related stresses in successive processes of semiconductor manufacturing with respect to high temperature or small impact in handling. To understand the mechanical properties of these thin film materials becomes more significant since the strength is likely to decrease while their size or thickness is getting smaller or thinner, resulting in the higher probability of failure or lower yield, and thus decreasing the device reliability. Some recent studies focused on integrated circuit metallization have noticed that the copper is the alternative material to replace aluminum because of its higher electrical and thermal conductivity for high speed applications. In general, the nature of the thin film materials and bulk materials are obviously different. For instance, the yield stress of polycrystalline thin film metal is higher than that of bulk material because the thin films regarding fine grain size restrict the motion of dislocation during deformation. Several researchers have studied the grain size effect on some materials [1–4]. Specifically speaking, Nieman et al. [1] present that the microhardness of nanocystalline Cu produced by inert gas condensation with fine grain size 3–50 nm is higher than that of coarse-grained Cu thin film. The yield strength of pure nanocrystalline Cu has six times higher than that of coarse-grained Cu thin film [2–4]. However, the mechanical behavior of thin copper film is rarely examined, especially for the study corresponding to different strain rates of mechanical loading. Previous studies have shown that the response of most engineering materials under dynamic loading is dramatically different from that of static conditions [5–7]. Although the flow stress along with the grain boundary increases with increasing strain rate, the actual effect of the strain rate is highly dependent on the nature of the tested material [8, 9]. For instance, some studies have proved the results by measuring the mechanical properties of bulk nanocrystalline metals [10–14]. The strain rates with a wide range (up to $10^3$ $s^{-1}$) to ultrafine grained Cu bulk materials indicated that the strain-rate sensitivity of ultra-fined grain metals was higher than that of annealed FCC polycrystalline metals [10, 11, 14]. However, the effect of strain rates to Cu thin film materials has remained unknown. In this study, the effect of strain rates for different thicknesses of Cu thin films deposited on thin polyimide foils was examined. The stress and strain of the test vehicle are measured by a micro tensile testing machine. Microstructure analysis was performed by X-ray diffraction meter to evaluate the grain size effect to the Cu thin film at various strain rates.

## 2. EXPERIMENT DETAILS

Polyimide foil (Kapton, DuPont) with a 125 μm thickness was used as substrate material. It can be deformed elastically to strains of more than 3%. Figure 1 presents the dimension of a test vehicle. It was fabricated to form a dog-bone shape with a parallel gage section of length 15 mm and width 5 mm. Prior to film deposition, the substrates were cleaned by rinsing with acetone and drying with pressurized Nitrogen gas. Later, the substrates were mounted in a sputter system (Duratek, Taiwan, base pressure of $5 \times 10^{-7}$ Torr). For the purposes of measuring deposited thickness, Si wafers were also used as substrates, films and Si wafers were deposited to have thicknesses in 500, 750, 1000 and 1500 nm. The deposited thickness and roughness on deposited wafers were measured by profile meter (Veeco, USA) respectively. The variation of measured thickness is less than $\pm 20$ nm. The grain sizes of the test vehicles were characterized using the X'Per Pro diffractometer (PANalytical, USA) with a Cu target. The width of the Bragg peaks corresponding to the dimension of coherent X-ray scattering was measured, and the average grain size was then calculated using the Scherrer equation [15]. The tensile tests were performed using a microforce test system (MTS® Tytron 250) machine. The strain rates were setup within a range of $1.6 \times 10^{-4}$ to $1.6 \times 10^{-2}$ $s^{-1}$. Figure 2 shows the relationship of load and strain of deposited Cu and PI film under a certain strain rate. It is seen that

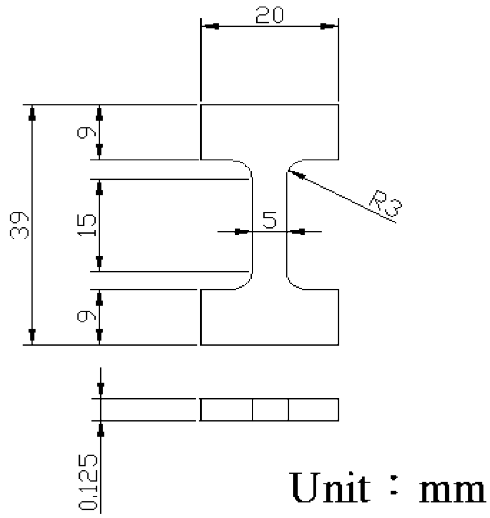


Fig. 1. Dimension of tensile test vehicle.

the loads of both specimen (PI and PI-Cu) monotonically increase while the strains of both them increase. Furthermore, note that the relationship between strain and load retains linearly within the 1% strain. As a result, we can infer that the loading of PI-Cu film is the sum of the loading applied to the PI and Cu films, respectively. Therefore, the stress of Cu thin film, $\sigma_{Cu}$, is shown as

$$\sigma_{Cu} = \frac{F_{total} - F_{PI}}{W_{Cu} \times t_{Cu}} \tag{1}$$

where $W_{Cu}$ and $t_{Cu}$ are the width and thickness of the Cu thin film respectively; $F_{total}$ and $F_{PI}$ are the force applied to the deposited Cu film on PI substrate and only PI substrate respectively. Assuming that the difference of the Poisson ratio between the two layers is quite small, it is neglected in this study [16].

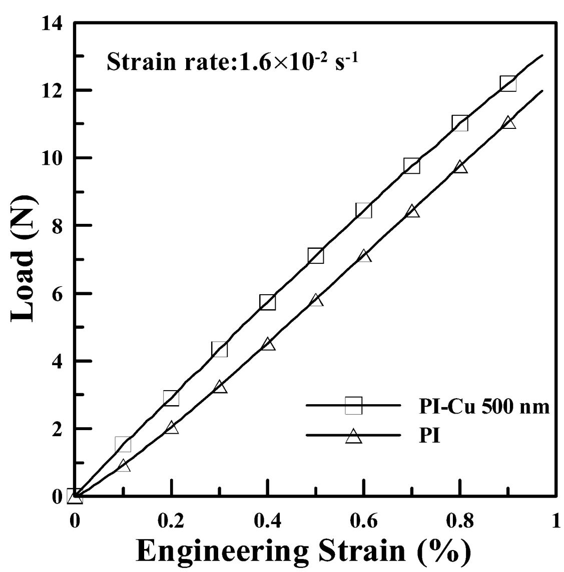


Fig. 2. The relationship of load and strain of deposited Cu and PI film under a strain rate condition.

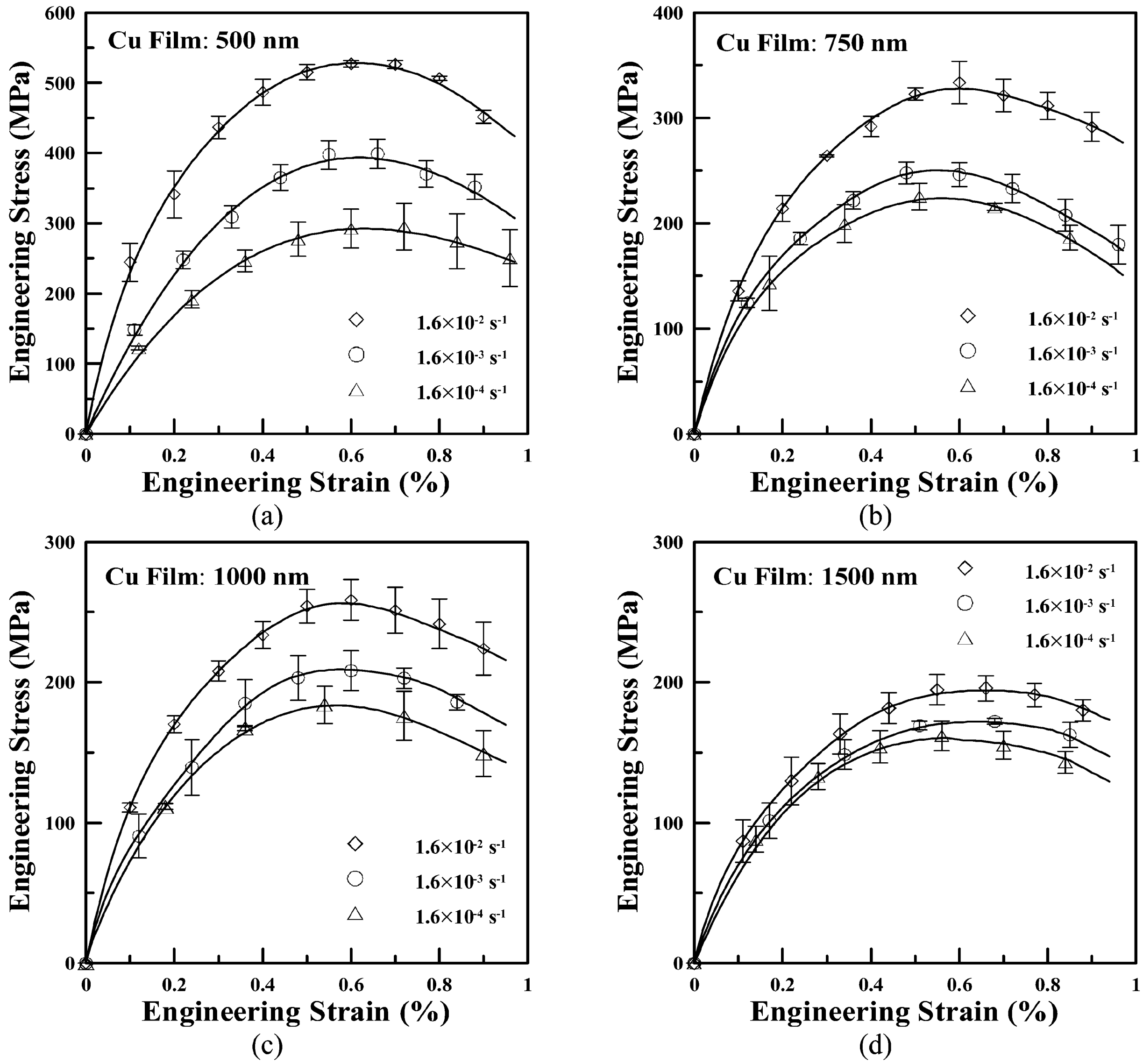


Fig. 3. The relationship of stress-strain of Cu thin film with (a) 500 nm; (b) 750 nm; (c) 1000 nm; (d) 1500 nm thickness under various strain rates.

## 3. RESULTS AND DISCUSSIONS

### 3.1. Stress-Strain Relationships

Figure 3 presents the relationship of stress-strain for Cu film with four different thicknesses. There are three strain rates $1.6 \times 10^{-4}$ , $1.6 \times 10^{-3}$ and $1.6 \times 10^{-2}$ $s^{-1}$ for each thickness of Cu thin film. It can be seen that the stress of Cu thin film increases with increasing strain rates, but the stress decreases with increasing the film thickness. Furthermore, the whole loading period is divided in two stages for all conditions: the first stage shows that the stress increases with increasing strain; the second one shows that the stress starts decreasing after the strain reaches a certain value until the specimen was broken. The results show that the initial cracks or flaws start propagating at a critical value of stress called max-stress [17], to reduce the strength of the material. In summary, the obvious difference of the stress-strain curves for various

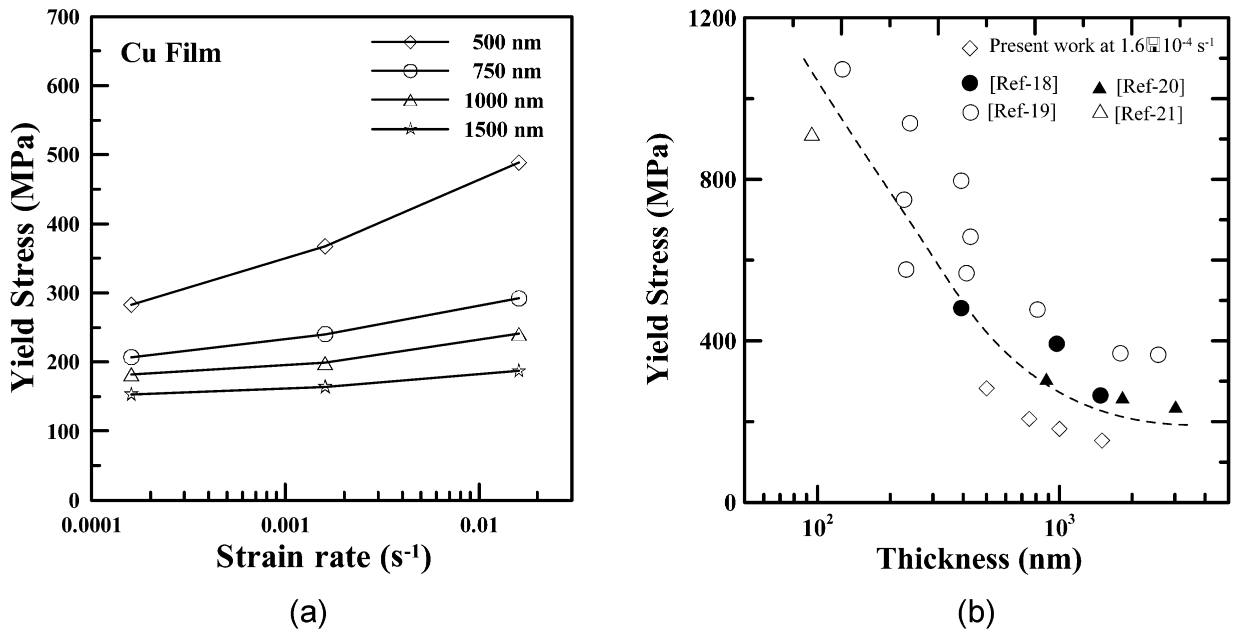


Fig. 4. (a) The relationship between the yield stress and the strain rate with various thicknesses of Cu films. (b). Comparison of the yield stress of Cu thin film. ◇ present work at $1.6\times10^{4}$ $s^{-1}$, • yield stresses measured by X-ray diffraction, ○ yield stresses tested on 12.5 µm thick PI substrates, ▲ yield stress measured by bulge test, △ yield stresses tested on 12.5 µm thick PI substrates.

thicknesses of Cu test vehicles indicates that the mechanical properties of Cu thin films are highly dependent on the strain rates.

### 3.2. Effect of Strain Rates

In order to analyze the effect of strain rates, some typical factors in mechanical behavior of the thin Cu films are selected, in particular, yield stress, Young's modulus, and maximum stress. Figure 4a illustrates the relationship between the yield stress and the strain rate as a function of the thicknesses of Cu films. The yield strength is defined as the stress that gives a plastic strain of 0.2%. It is seen that the yield stress increases as the strain rate increases for all thicknesses of Cu films. Meanwhile, the thinner film has a higher yield stress. Figure 4b shows the comparison of using various test methods [18–21] to evaluate the yield strength of Cu thin films. Compared to other studies, the results of our methodology represent the same trend for the thickness effect in yield strength. As the strain rate increases, the yield stress increases by 206 and 34 MPa for the thinnest (500 nm) and the thickest (1500 nm) Cu film, respectively, resulting in the fact that thinner Cu film may have higher sensitivity to the various strain rates. There is the same result with the Young's modulus, shown in Fig. 5. Therefore, a thinner Cu film is capable of having a large strengthening effect under dynamic loading. Figure 6 shows the relationship between the maximum stress and the thicknesses of Cu films with various strain rates. The maximum stress $\sigma_{\max}$ is close to 550 MPa occurred at the combinations of the thinnest film (500 nm) and the fast strain rate ($1.6\times10^{-2}$ $s^{-1}$). There is about 2.75 times the maximum stress of the thickest (1500 nm) film under the same strain rate, resulting in a higher failure probability if the size of flaws (cracks) in these two thicknesses is similar since it is generally seen that the material was fabricated by the same process.

Alternatively, the relationship between the yield stress and the strain rate for the Cu thin film is given by

$$\sigma_y = c\dot{\varepsilon}^m|_{\varepsilon,T} \tag{2}$$

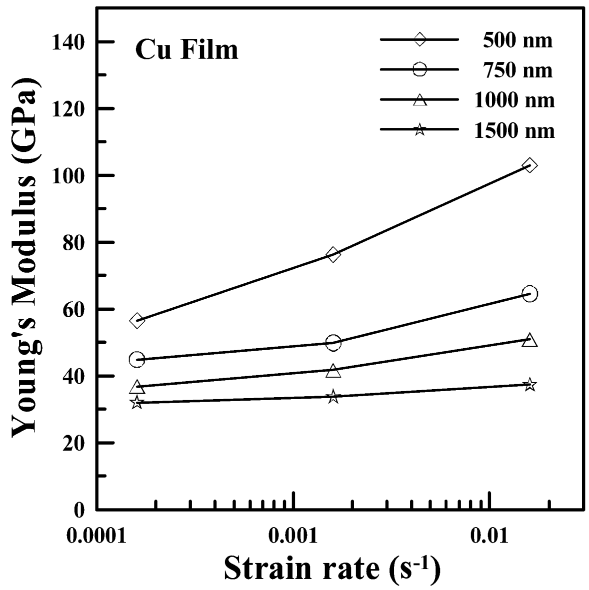


Fig. 5. The relationship between the Young's modulus and the strain rate with various thicknesses of Cu films.

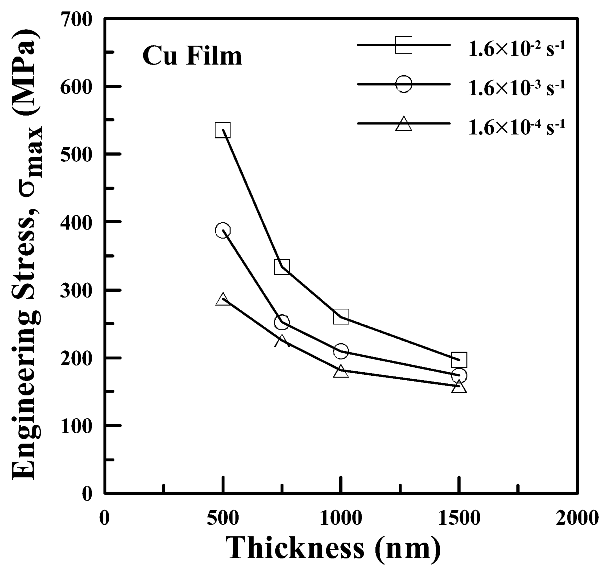


Fig. 6. The relationship between the maximum stress and the thicknesses of Cu films t with various strain rates.

where $\sigma_y$ is the yield stress; $\dot{\varepsilon}$ is the strain rate; $c$ is a material constant, and $m$ is the strain rate sensitivity of the yield stress. By taking the logarithm of both sides of equation (2), we obtain

$$\ln \sigma_y = m \ln \dot{\varepsilon} + \ln c \tag{3}$$

Figure 7 presents the logarithmic plot of relationship between the yield stress and the strain rate with various thicknesses of Cu films. The strain rate sensitivity $m$ of the yield stress is 0.119, 0.075, 0.061, and 0.043 with Cu film thicknesses of 500, 750, 1000, and 1500 nm respectively. The values of $m$ decrease as the thickness of Cu film increases. Compared to the bulk Cu material at room temperature, the values range from 0.006 to 0.012 [22, 23]. It can be concluded that the Cu thin film is more sensitive in various strain

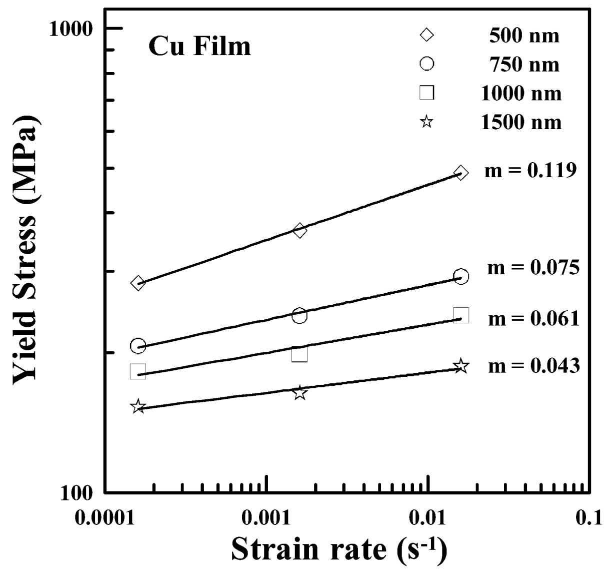


Fig. 7. The logarithmic plot of the relationship between the yield stress and the strain rate with various thicknesses of Cu films.

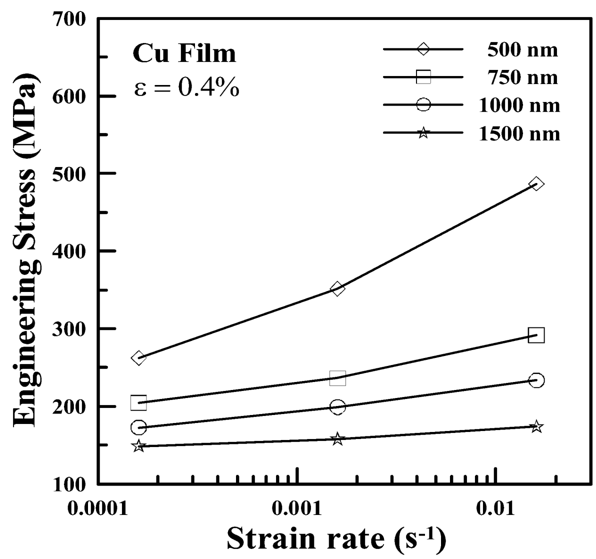


Fig. 8. Variations of shear stress with strain rate at a strain of 0.004 under different thicknesses of Cu film conditions.

rates than bulk Cu material. Therefore, it should be more careful to handling the thin Cu materials when they are in fabricating or in use conditions.

The alternative study of effect in strain rate and thickness of Cu thin film during the mechanical loading is to estimate the shear stress as a function of the logarithmic strain rate. Figure 8 shows a variation of shear stresses with strain rates at a strain of 0.004 using different thicknesses of Cu film. For each thickness of Cu film, it can be seen that the shear stress increases as the strain rate increases. In addition, for a certain strain rate, the shear stress increases as the thickness of Cu film decreases. Furthermore, the stress induced

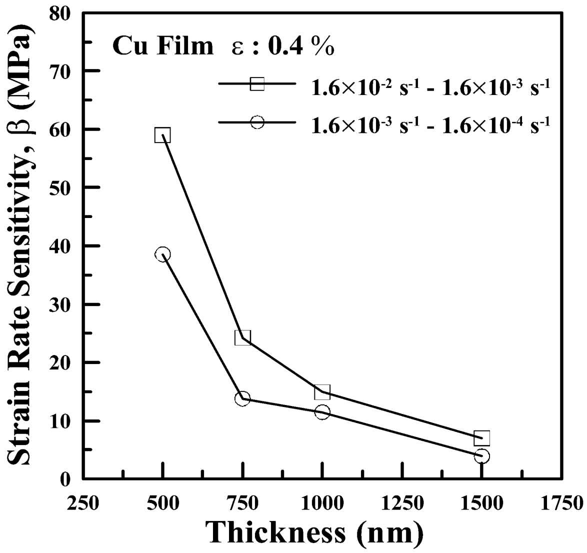


Fig. 9. Variations of the strain rate sensitivity parameter $\beta$ with the strain as a function of the thickness of Cu film and strain rate.

by an increased strain rate at various thicknesses can be quantified using the following strain rate sensitivity parameter $\beta$ [24]

$$\beta = \frac{\sigma_2 - \sigma_1}{\ln(\dot{\varepsilon}_2 - \dot{\varepsilon}_1)} \tag{4}$$

where the stress values $\sigma_2$ and $\sigma_1$ are obtained in tests conducted at constant strain rates of and respectively, and they are calculated at the same value of the strain. Figure 9 illustrates the variation of the strain rate sensitivity parameter $\beta$ with the strain as a function of the thickness of Cu film and strain rate. It is seen that, for a given thickness, the strain rate sensitivity increases with increasing the strain rate. Furthermore, the strain rate sensitivity rapidly increases as the thickness decreases from 750 to 500 nm. Thus, it implies that the workhardening rate increases while the thickness of Cu film decreases, resulting in a higher probability of brittle failure.

### 3.3. Grain Size Strengthening

Grain size is one of the critical factors in the strengthening mechanism associated with yield stress. According to the measurement of X-ray diffraction, the grain size for each thickness of Cu film is identified. Figure 10 shows the relationship between gain size and the thickness of Cu film. It indicated that the grain size is a linear function of film thickness using X-ray diffraction. Grain size grows in thicker Cu film. Further it can be noted the dependence of yield stress on grain size follows the Hall–Petch equation [25–29]:

$$\sigma_y = \sigma_0 + kD^{-1/2} \tag{5}$$

where $\sigma_y$ is the yield stress; $\sigma_0$ is the frictional stress required to move dislocations, and $D$ is the grain size. Figure 11 reveals variations of the yield stress as a function of grain size $D^{-1/2}$ under different strain rates. It is seen that the yield stress parabolically increases as the grain size increases with $D^{-1/2}$ for all strain rate conditions. The yield stress dramatically increases for a finer grain size of the Cu film, especially in the fast strain rate of $1.6 \times 10^{-2}$ s$^{-1}$. From the aspect of microstructure, it can be inferred that more dislocations of pile-up or initiation through grain boundary speed up the increase of yield stress at a higher strain rate during mechanical loading.

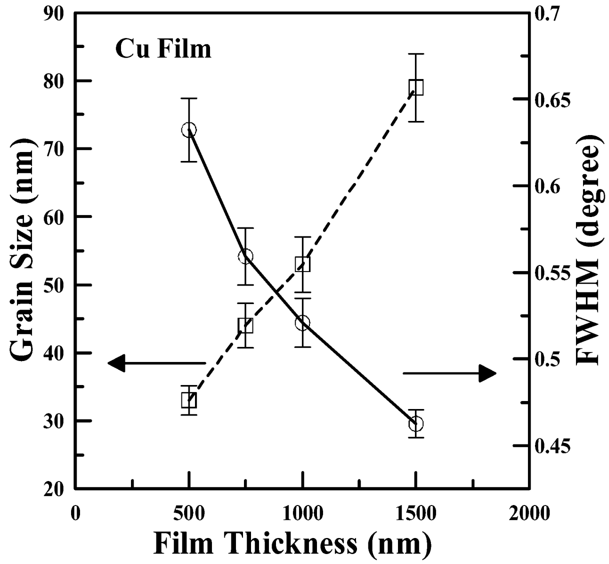


Fig. 10. The relationship between gain size and the thickness of Cu film.

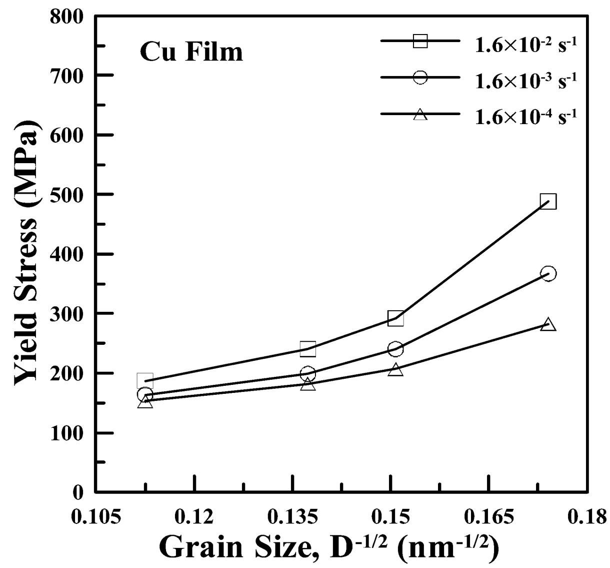


Fig. 11. Variations of the yield stress as a function of grain size $D^{-1/2}$ under different strain rate conditions.

## 4. CONCLUSIONS

The tensile tests combined with different strain rates and thicknesses of Cu film were performed. The results show that the strain rate and the thickness of test vehicle have obvious influence upon the mechanical properties of Cu thin film. The selected factors corresponding to yield stress, Young's modulus, and maximum tensile stress increase with increasing the strain rate or decreasing the thickness of the Cu film. Considering the strain rate sensitivity, it rapidly increases as the thickness from 750 to 500 nm decreases, to imply that the workhardening rate increases while the thickness of Cu film decreases, resulting in a higher probability of brittle failure. From the aspect of microstructure, it can be inferred that more dislocations of pile-up or initiation through grain boundary speed up the increase of yield stress at a higher strain rate during mechanical loading.

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## REFERENCES

1. Nieman, G.W., Weertman, J.R. and Siegel, R.W., "Mechanical behavior of nanocrystalline Cu and Pd", *Journal of Materials Science*, Vol. 6, pp. 1012–1027, 1991.
2. Koch, C.C., Morris, D.G., Lu, K. and Inoue, A., "Ductility of nanostructured materials", *Materials Research Bulletin*, Vol. 24, pp. 54–58, 1999.
3. Weertman, J.R., Farkas, D., Hemker, K., Kung, H., Mayo, M., Mitra, R. and Van Swygenhoven, H., "Structure and mechanical behavior of bulk nanocrystalline materials", *Materials Research Bulletin*, Vol. 24, pp. 44–50, 1999.
4. Sanders, P.G., Youngdahl, C.J. and Weertman, J.R., "The strength of nanocrystalline metals with and without flaws", *Materials Science and Engineering A*, Vols. 234–236, pp. 77–82, 1997.
5. Seidensticker, J.R. and Mayo, M.J., "Dynamic and static grain growth during the superplastic deformation of 3Y-TZP", *Scripta Materialia*, Vol. 38, pp. 1091–1100, 1998.
6. Sedighiand, M. and Mohammadi, B., "On the static and dynamic analysis of a small satellite (MESBAH)", *Acta Astronautica*, Vol. 52, pp. 1007–1012, 2003.
7. Hsu, C.H. and Teng, H.Y., "Temperature effects on the static and dynamic fracture behaviors of low-silicon CA-15 tempered stainless steel castings", *Journal of Nuclear Materials*, Vol. 340, pp. 1–11, 2005.
8. Butcher, B.M. and Karnes, C.H., "Strain rate effects in metals", *Journal of Applied Physics*, Vol. 37, pp. 402–411, 1966.
9. Wei, Q., Cheng, S., Ramesh, K.T. and Ma, E., "Effect of nanocrystalline and ultrafine grain sizes on the strain rate sensitivity and activation volume: fcc versus bcc metals", *Materials Science and Engineering A*, Vol. 381, Nos. 1–2, pp. 71–79, 2004.
10. Gray, G.T. III, Lowe, T.C., Cady, C.M., Valiev, R.Z. and Aleksandrov, I.V., "Influence of strain rate & temperature on the mechanical response of ultrafine-grained Cu, Ni, and Al-4Cu-0.5 Zr", Nanostructured Materials, Vol. 9, No. 1, pp. 477–480, 1997.
11. Wang, N., Wang, Z.R., Aust, K.T. and Erb, U., "Room temperature creep behavior of nanocrystalline nickel produced by an electrodeposition technique", *Materials Science and Engineering A*, Vol. 237, pp. 150–158, 1997.
12. Mayo, M.J., Siegel, R.W., Narayanasamy, A. and Nix, W.D., "Mechanical properties of nanophase $TiO_2$ as determined by nanoindentation", *Journal of Materials Research*, Vol. 5, pp. 1073–1082, 1990.
13. Mayo, M.J., Siegel, R.W., Liao, Y.X. and Nix, W.D., "Nanoindentation of nanocrystalline ZnO", *Journal of Materials Research*, Vol. 7, pp. 973–979, 1992.
14. Cheng, S., Ramesha, K.T. and Ma, E., "Effect of nanocrystalline and ultrafine grain sizes on the strain rate sensitivity and activation volume: fcc versus bcc metals", *Materials Science and Engineering A*, Vol. 381, Nos. 1–2, pp. 71–79, 2004.
15. Klug, H.P. and Alexander, L.E., *X-Ray Diffraction Procedure: For Polycrystalline and Amorphous Materials*, 2nd edn., Wiley-VCH, 1974.
16. Macionczyk, F., Brückner, W., Pitschke, W. and Reiss, G., "Young's modulus and tensile strength of CuNi(Mn) thin films on polyimide foils by tensile testing", *Journal of Materials Research*, Vol. 13, No. 10, pp. 2852–2858, 1998.
17. Macionczyk, F. and Bruckner, W., "Tensile testing of AlCu thin films on polyimide foils", *Journal of Applied Physics*, Vol. 86, pp. 4922–4929, 1999.
18. Hommel, M. and Kraft, O., "Deformation behavior of thin copper films on deformable substrates", *Acta Materialia*, Vol. 49, pp. 3935–3947, 2001.
19. Yu, Denis Y.W. and Spaepen, F., "The yield strength of thin copper films on Kapton", *Journal of Applied Physics*, Vol. 95, No.6, pp. 2991–2997, 2004.

20. Xiang, Y., Chen, X. and Vlassakv, J.J., “The mechanical properties of electroplated Cu thin films measured by means of the bulge test technique”, *Materials Research Society Symposium Proceedings*, Vol. 695, L.4.9.1–L.4.9.6, 2002.
21. Zhang, B., Sun, K.H., Gong, J., Sun, C., Cai Q.K., and Zhang, G.P., “Tensile properties of 100 nm thick Cu films”, *Chinese Journal of Materials Research*, Vol. 20, No. 1, pp. 29–32, 2006.
22. Follansbee, P.S., Regazzoni, G. and Kocks, U.F., “Mechanical properties at high rates of strain”, *Journal of Physics: Conference Series*, Vol. 70, pp. 71–80, 1984.
23. Follansbee, P.S. and Kocks, U.F., “A constitutive description of the deformation of copper based on the use of the mechanical threshold stress as an internal state variable”, *Acta Materialia*, Vol. 36, pp. 81–93, 1988.
24. Ezz, S.S., Sue, Y.Q. and Hirsch, P.B., “Strain rate dependence of the flow stress and work hardening of $\gamma'$”, *Materials Science and Engineering A*, Vols. 192–193, pp. 45–52, 1995.
25. Hall, E.O., “The deformation and ageing of mild steel: III Discussion of results”, in *Proceedings of the Physical Society*, London, UK, B64, pp. 747–752, 1951.
26. Petch, N.J., “The cleavage strength of polycrystals”, *The Journal of the Iron and Steel Institute*, Vol. 174, pp. 25–28, 1953.
27. Weertman, J.R., “Hall–Petch strengthening in nanocrystalline metals”, *Materials Science and Engineering: A*, Vol. 166, Nos. 1–2, pp. 161–167, 1993.
28. Tong, C.J., Cheng, Y.C., Lin, M.T., Chung, K.J., Hsu, J.S. and Wu, C.L., “Optical micro-peddle beam deflection measurement for electrostatic mechanical testing of nano-scale thin film application to MEMS”, *Microsystem Technologies*, Vol. 16, No. 7, pp. 1131–1137, 2010.
29. Chen, Y.T., Chung, K.J., and Lin M.T., “Mechanical property measurement of nano-scale argentum films on the novel paddle cantilever beams using four step phase-shifting method”, in *Proceedings of IEEE International Conference on Electronics Materials and Packaging (EMAP'08) – The 3rd International Microsystems, Packaging, Assembly and Circuits Technology (IMPACT) Conference and the 10th International Conference on Electronics Materials and Packaging (EMAP)*, pp. 179–182, 2008.