Deformation mechanism of amorphous/nanocrystalline multilayers thin films on polyimide substrates
奈米與非晶多層薄膜於聚亞醯胺基板上之變形機制

Student: H. S. Huang

Adviser: Prof. J. C. Huang

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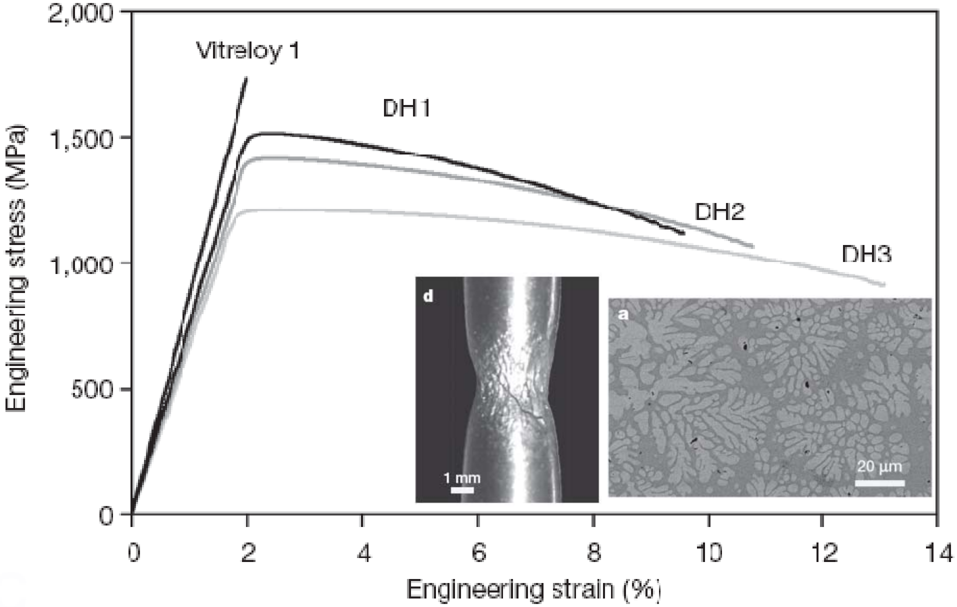
Improvement of tensile ductility for metallic glasses

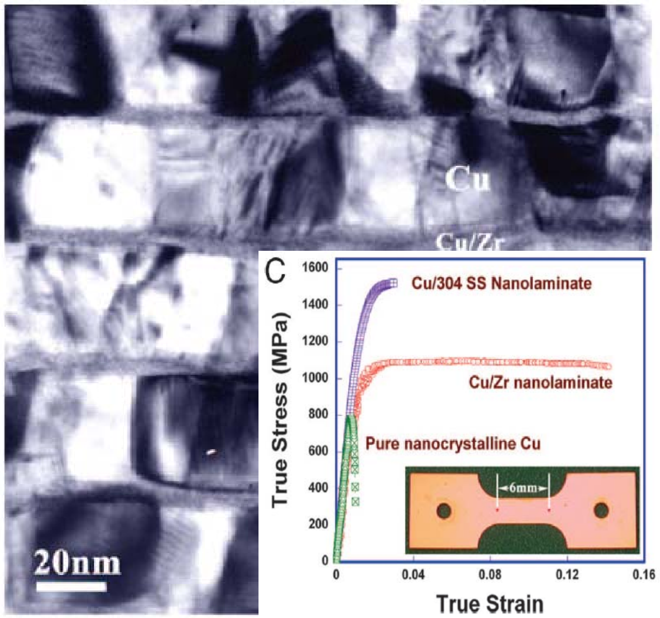
Drawbacks of metallic glass: **Tensile brittleness**

Methods of improvement:

1. **In-situ or ex-situ composites**
2. **Multilayer**

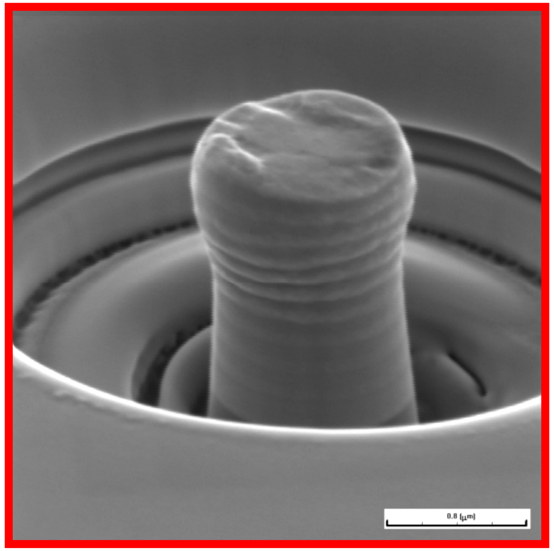


D. C. Hafmann et al. Nature (2008)

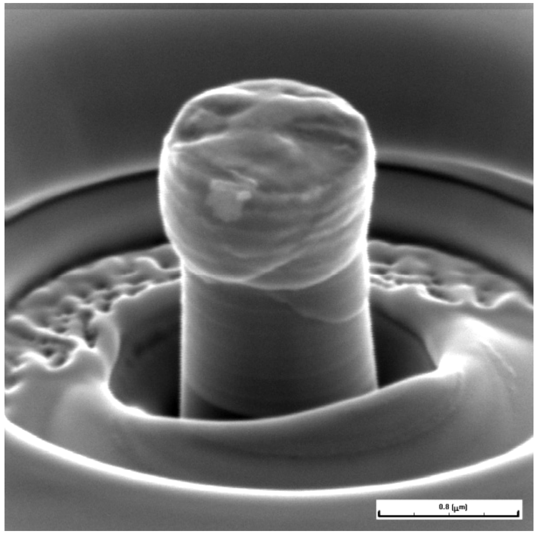


Y. Wang et al. Proc. Natl. Acad. Sci. USA (2007)

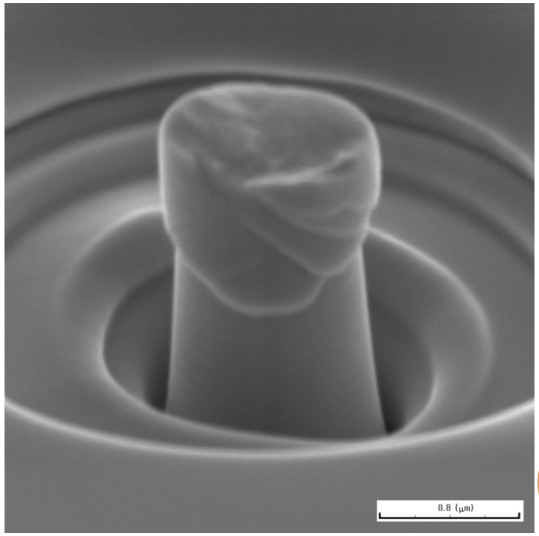
Compression tests of multilayered thin film metallic glass



ZrCu/Cu (100 nm/100 nm)



ZrCu/Cu (100 nm/50 nm)



ZrCu/Cu (100 nm/10 nm)

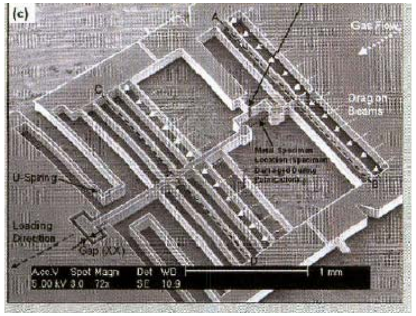
Metallic glass based composites (MGCs)

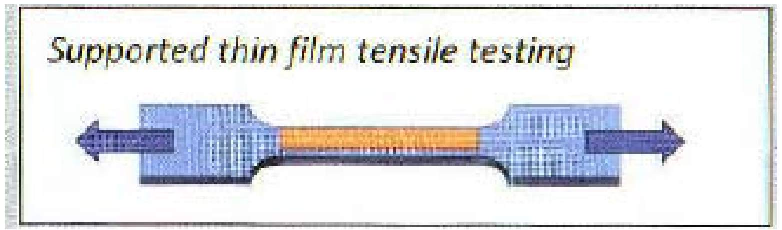
I. C. Lin, Master Thesis (2010).

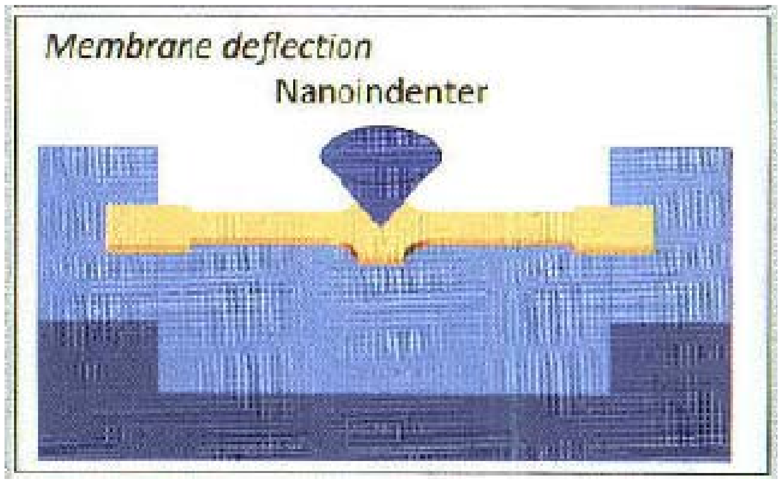
Tensile test of thin film

Method of tensile test for thin films:

1. **Tension system integrated in MEMS**
2. **Single tensile specimen without MEMS**
3. **Supported thin film tensile testing**

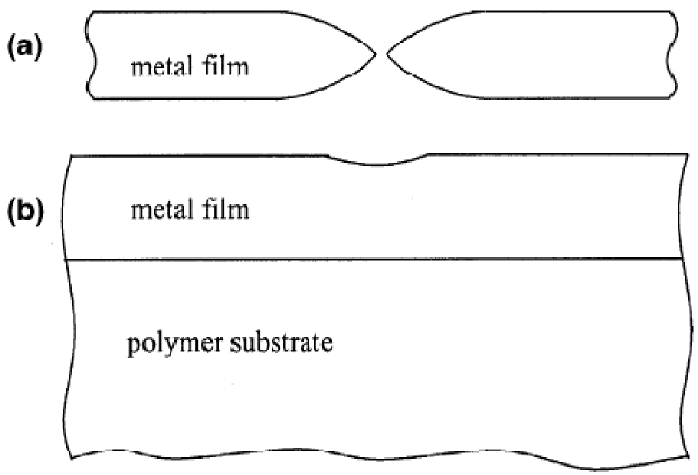






Influence factors of supported thin film tests

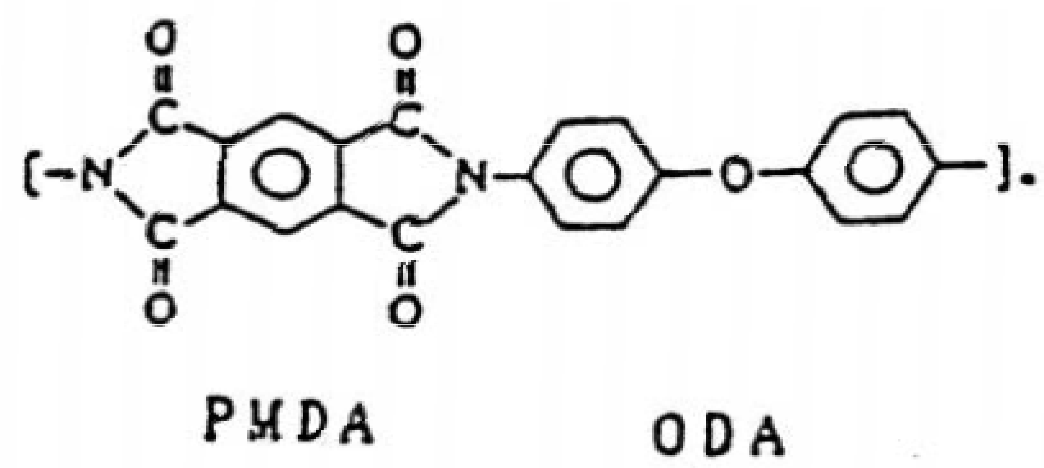
1. **Substrate materials**
2. **Thickness of substrates**
3. **Roughness of substrates**
4. **Adhesion between films and substrates**



T. Li et al. Mech. Mat. (2005)

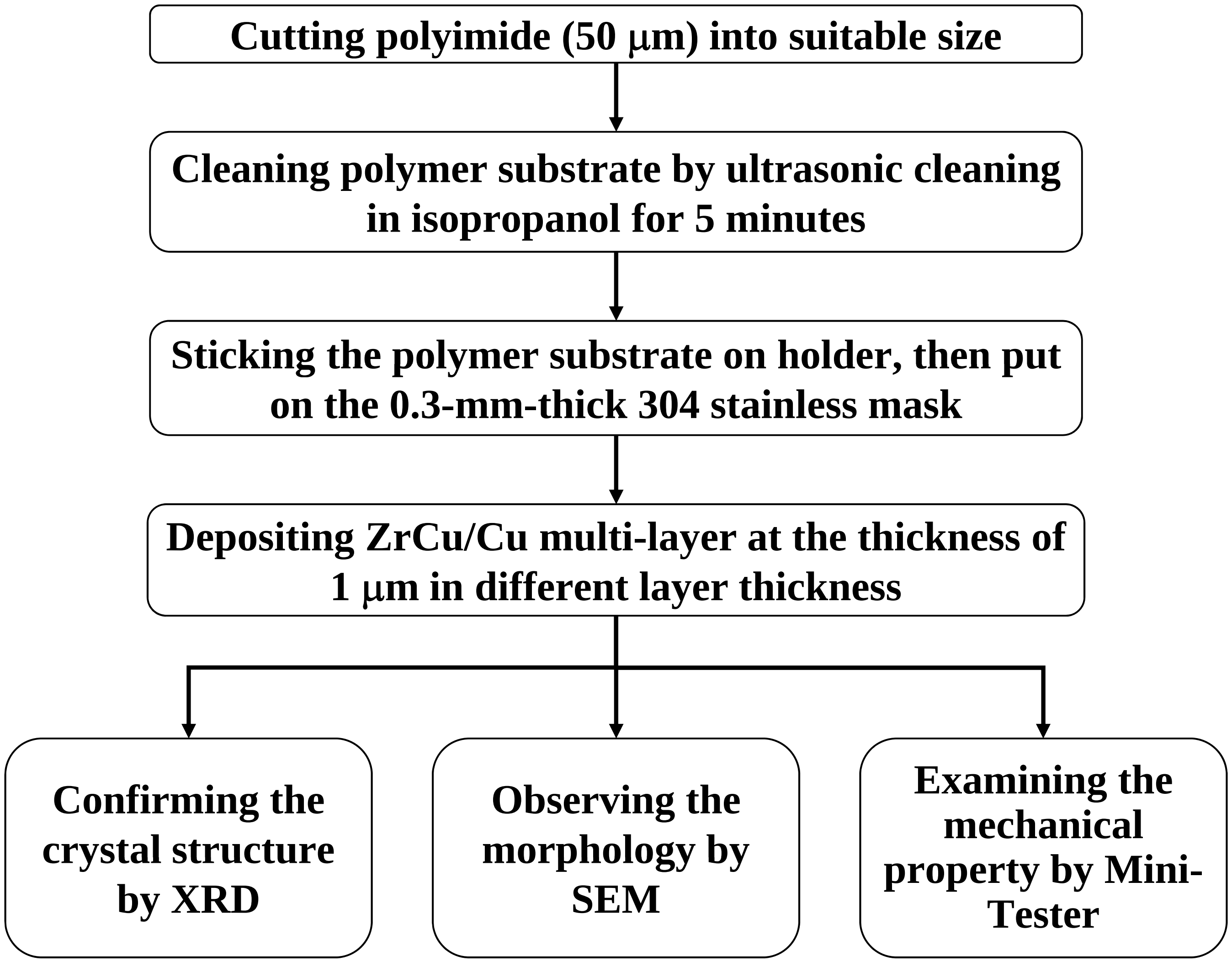
Properties of polyimide (Kapton®)

- **Thermal property: High glass transformation temperature of 360 - 410 °C**
- **Mechanical properties: High yield strength, toughness and fracture stress**



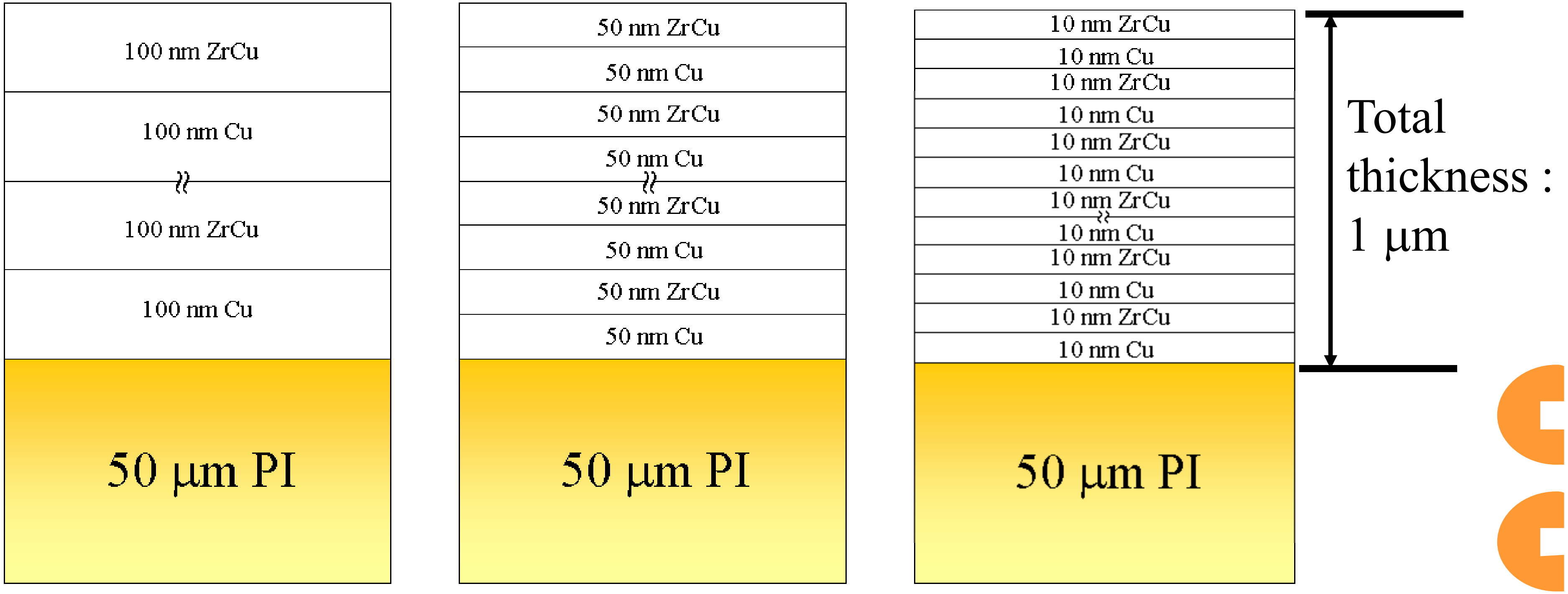
S. Iwamori et al. Vacuum (1998)

Flow chart of the experimental procedure



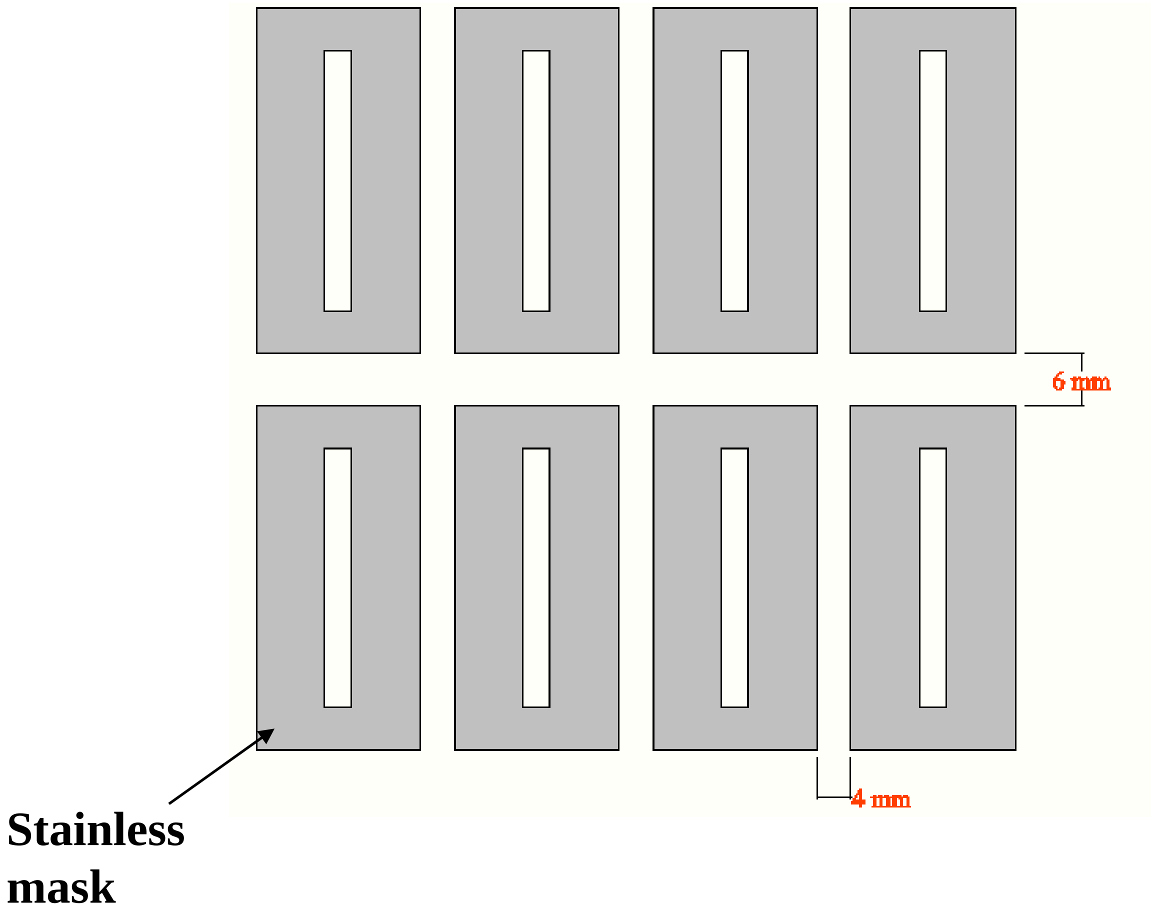
Schematic representation of specimen dimensions

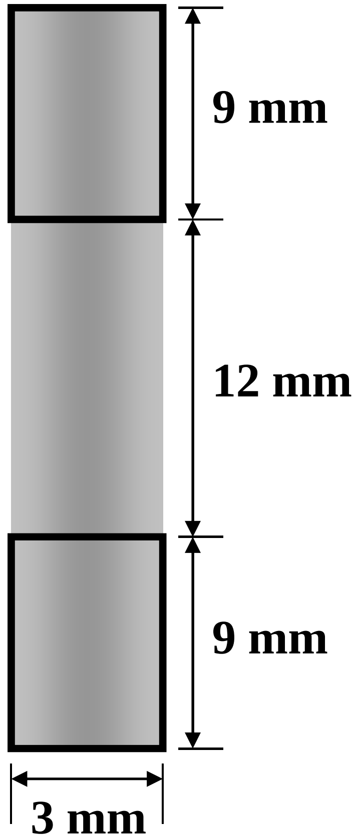
Thin film deposition: sputtering



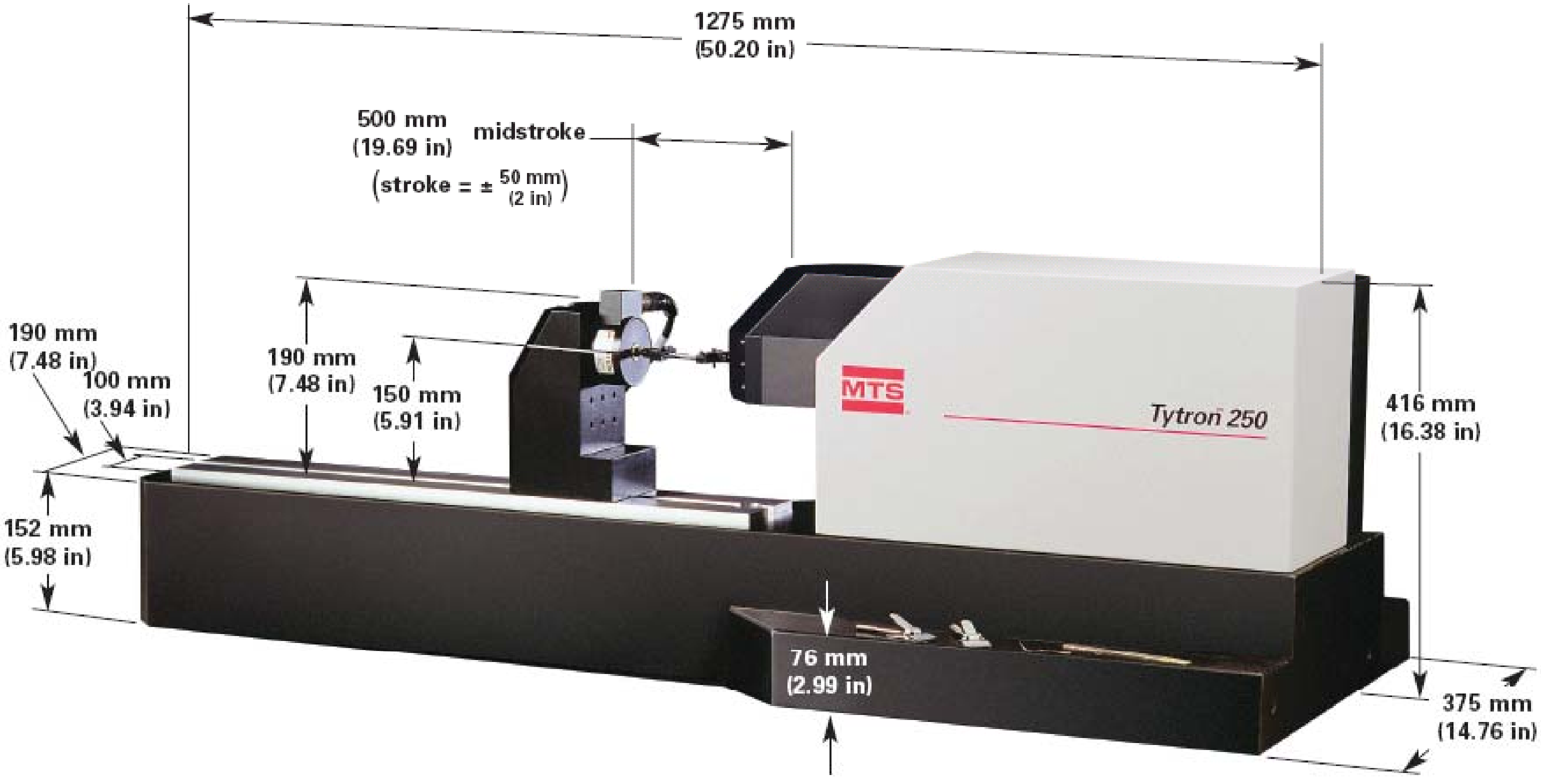
Layer thicknesses: 100 nm/100 nm, 50 nm/50 nm, 25 nm/25 nm, 10 nm/10 nm

Schematic representation of specimen dimensions



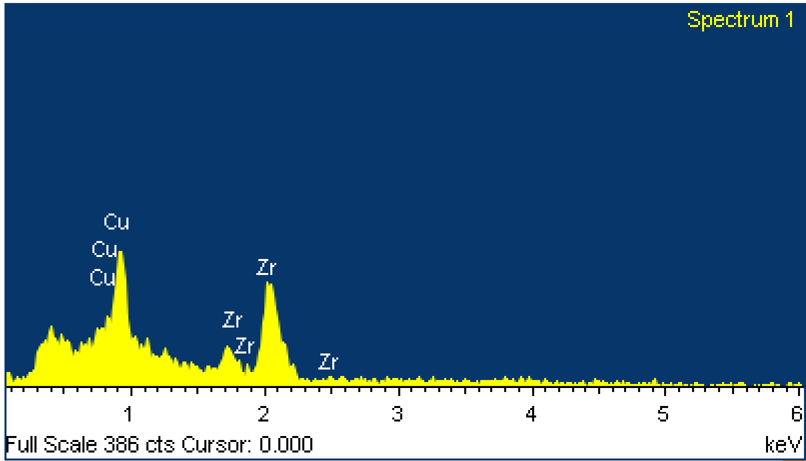


MTS Tytron™ 250 Microforce Testing System



EDS results

	Zr (at. %)	Cu (at. %)
1	53.63	46.37
2	52.54	47.46
3	53.08	46.92
4	53.63	46.37
Avg.	53.22	46.78

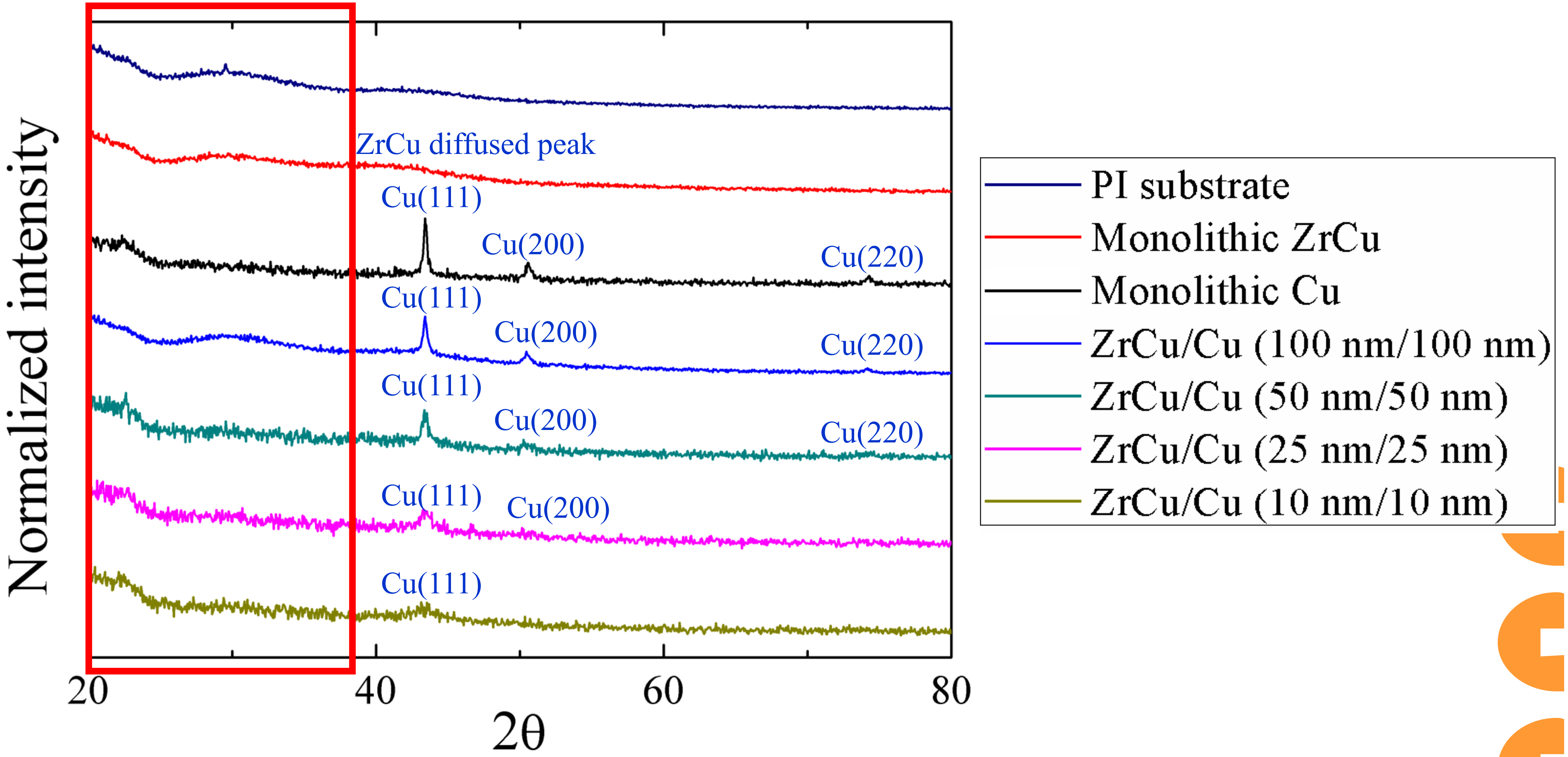


Close to setting goal: $Zr_{50}Cu_{50}$

Outline

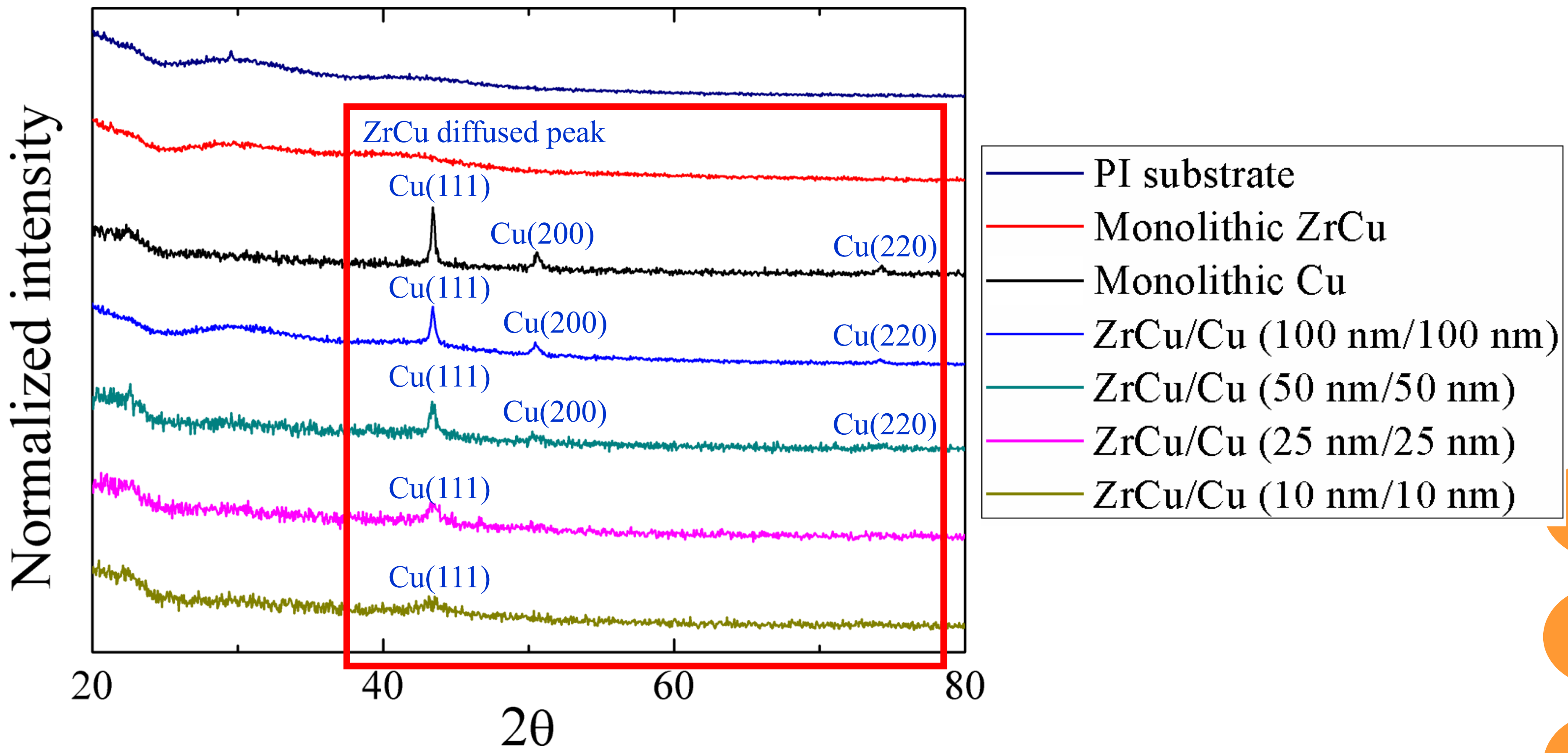
1. Introduction and motivation
2. Experimental procedures
3. **Results and discussion**
    1. SEM/EDS analysis of the ZrCu sample
    2. **XRD analysis**
    3. SEM film surface morphology characterization
    4. Mechanical analysis
    5. SEM fracture surface morphology analysis
4. Conclusion

XRD results



Diffraction peaks of low angles are contributed by the PI substrate.

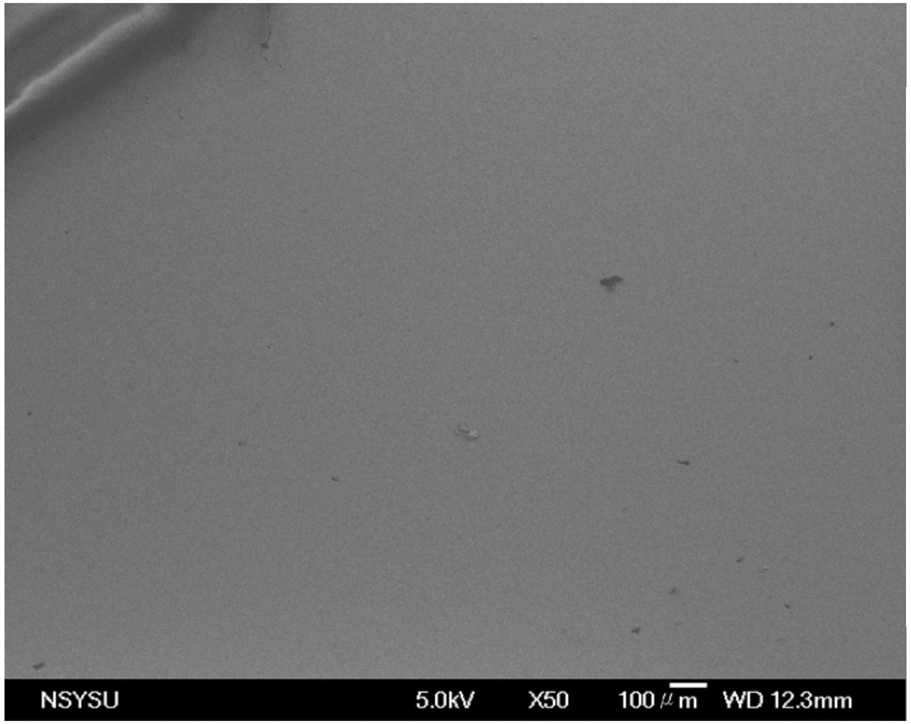
XRD results

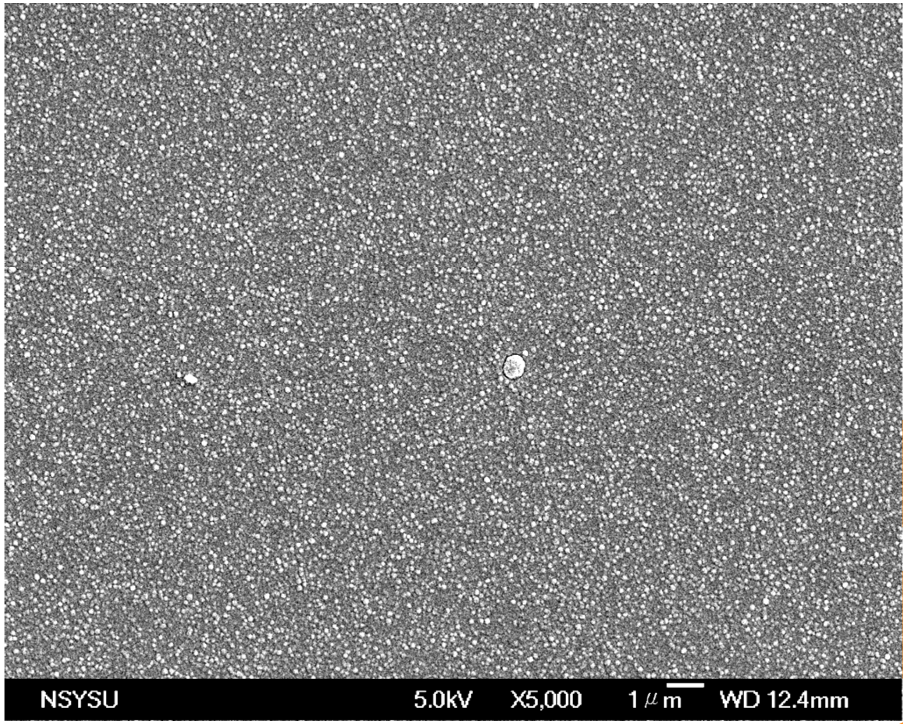


As the layer thickness getting lower, the height of characteristic peaks of Cu become lower and closer to amorphous ZrCu diffused peak.

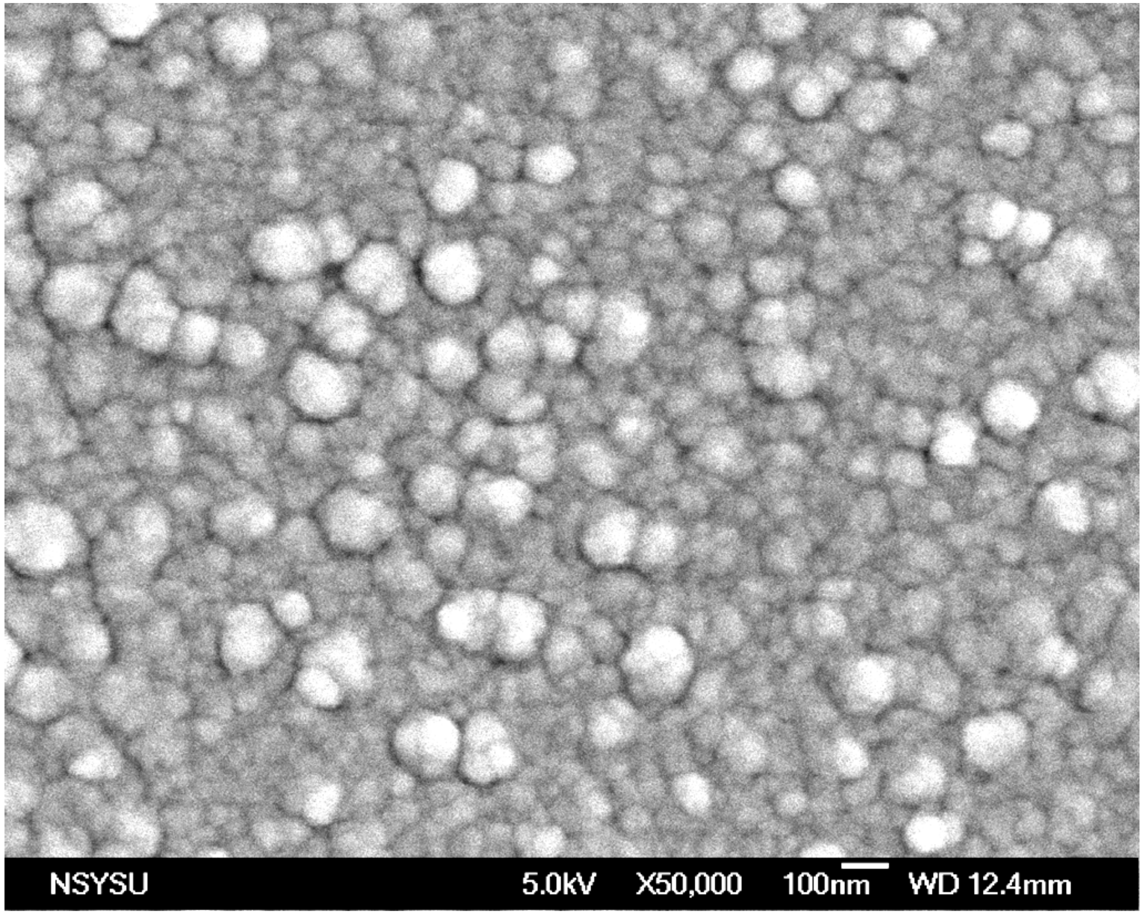
SEM morphology

ZrCu/Cu (100 nm/100 nm)

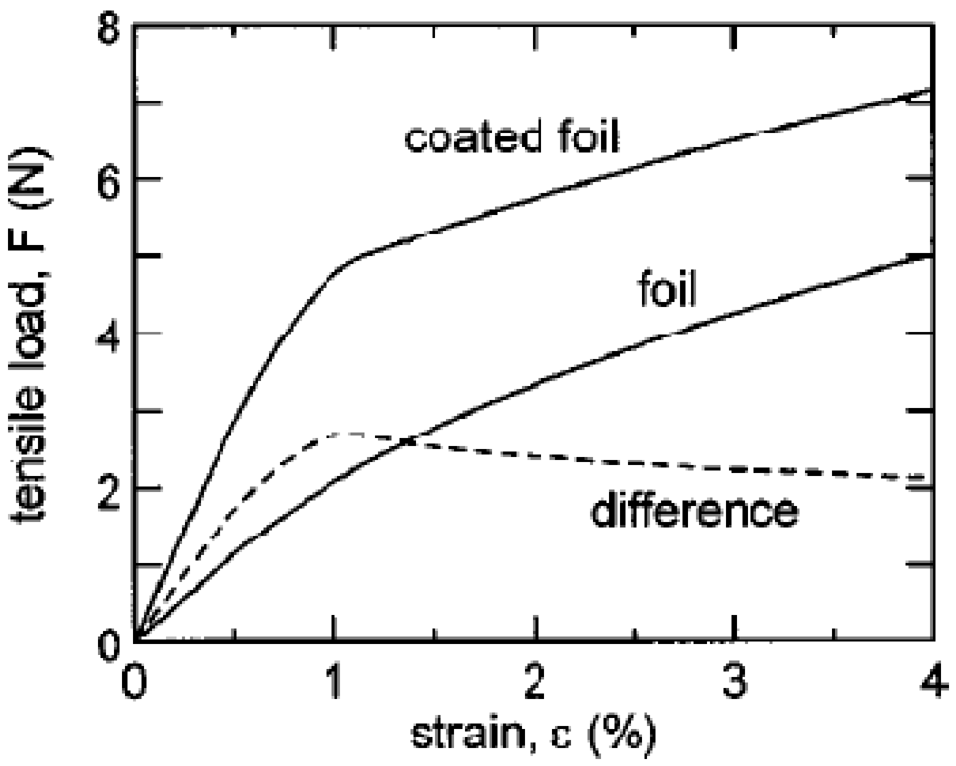




SEM morphology



Data extraction

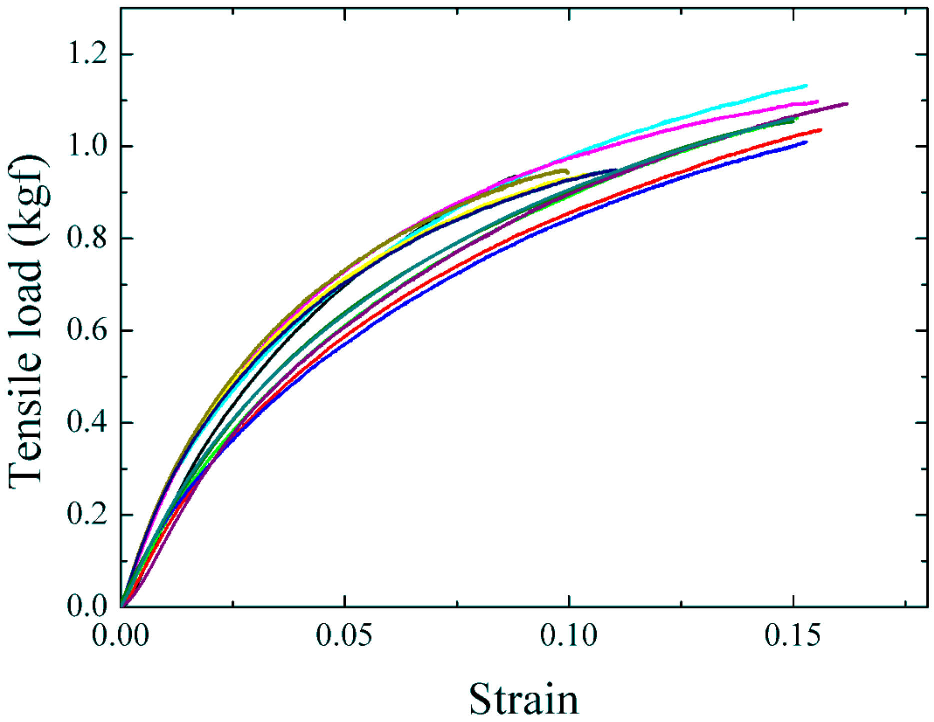


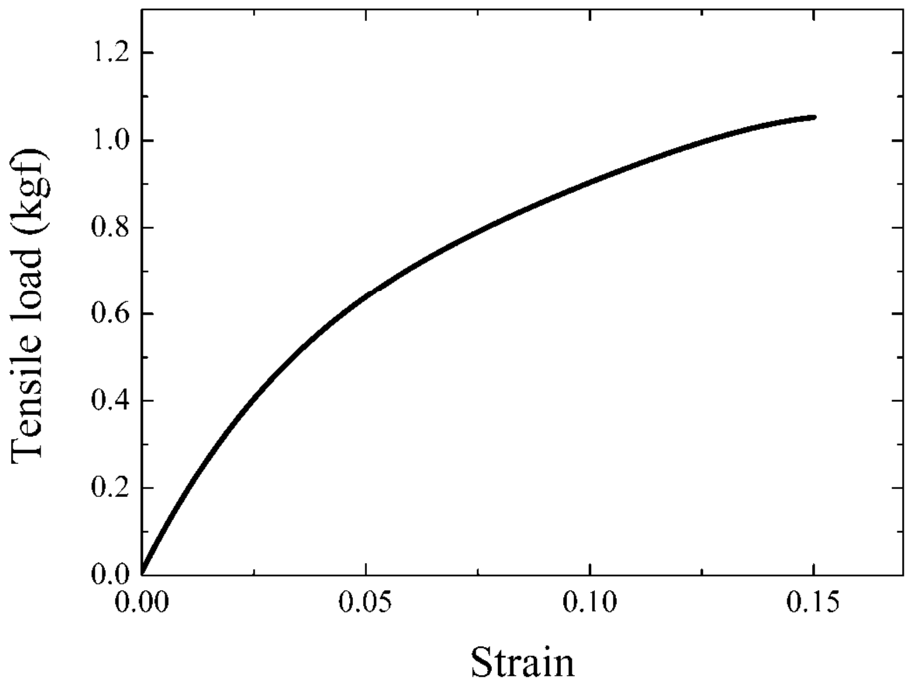
The stress in the film is obtained by **deducting** the contribution of the **substrate** from the **total force**:

$$\sigma_{film} = \frac{1}{w_{film} t_{film}} (F_{total} - F_{Kpton})$$

F. Macionczyk and W. Brűckner, J. Appl. Phys. (1999).

Data fitting of PI substrate

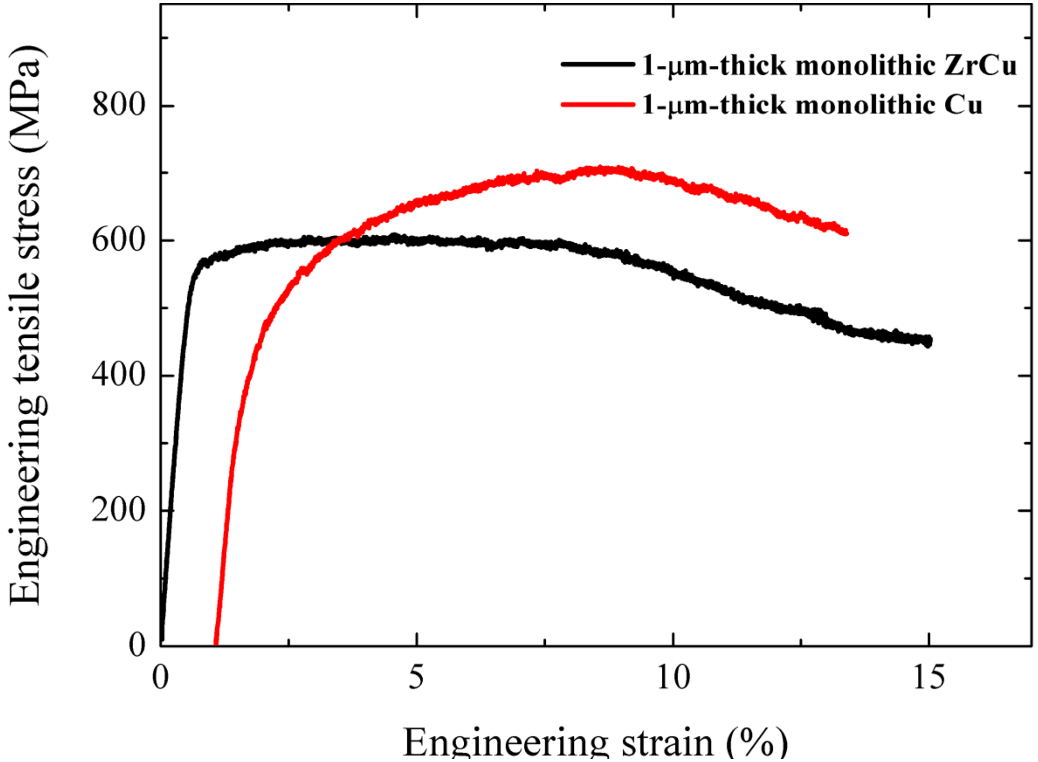




All lines were averaged and fitted by 4-order equation:

$$F = 12.67112\varepsilon - 137.88107\varepsilon^2 + 875.12079\varepsilon^3 - 2214.3863\varepsilon^4$$

Comparison of ZrCu and Cu thin films



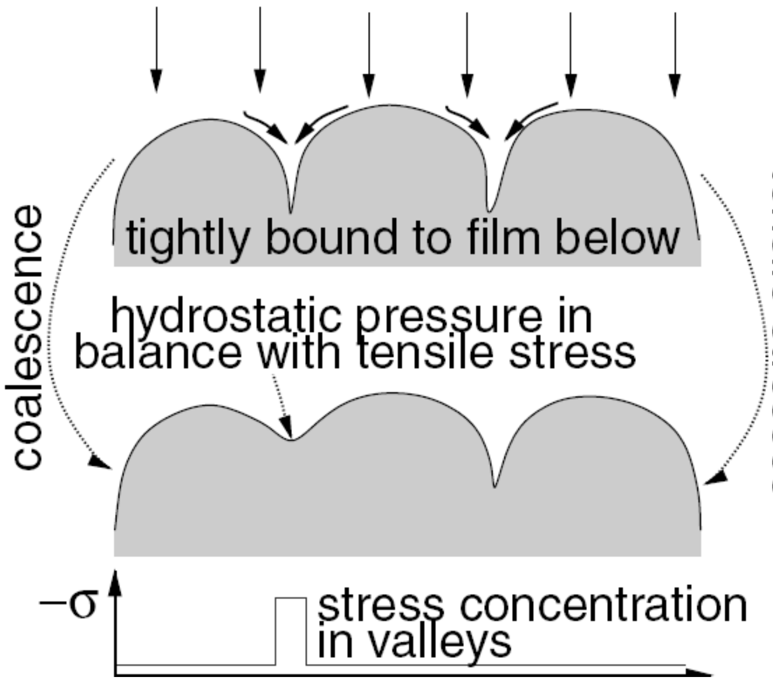
Rule of mixture (ROM) in iso-strain type:

$$E_{multilayer} = (1 - f) \times E_{ZrCu} + f \times E_{Cu}$$

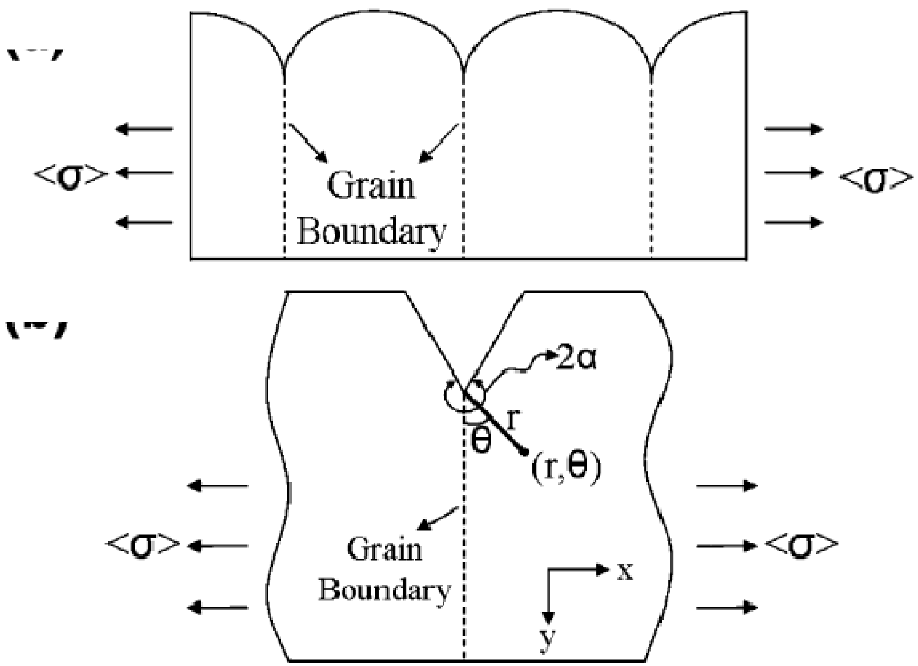
	Testing methods	Young's modulus (GPa)
Amorphous ZrCu	Tensile test	83
Crystalline Cu		104
Theoretical ROM values		93
Amorphous ZrCu	Nano-indentation	93
Crystalline Cu		127
Theoretical ROM values		110

M. C. Liu et al., Scripta Mater. (2009)
H.D. Espinosa et al., J. Mech. Phys. Solids (2004)

Comparison of monolithic metal films

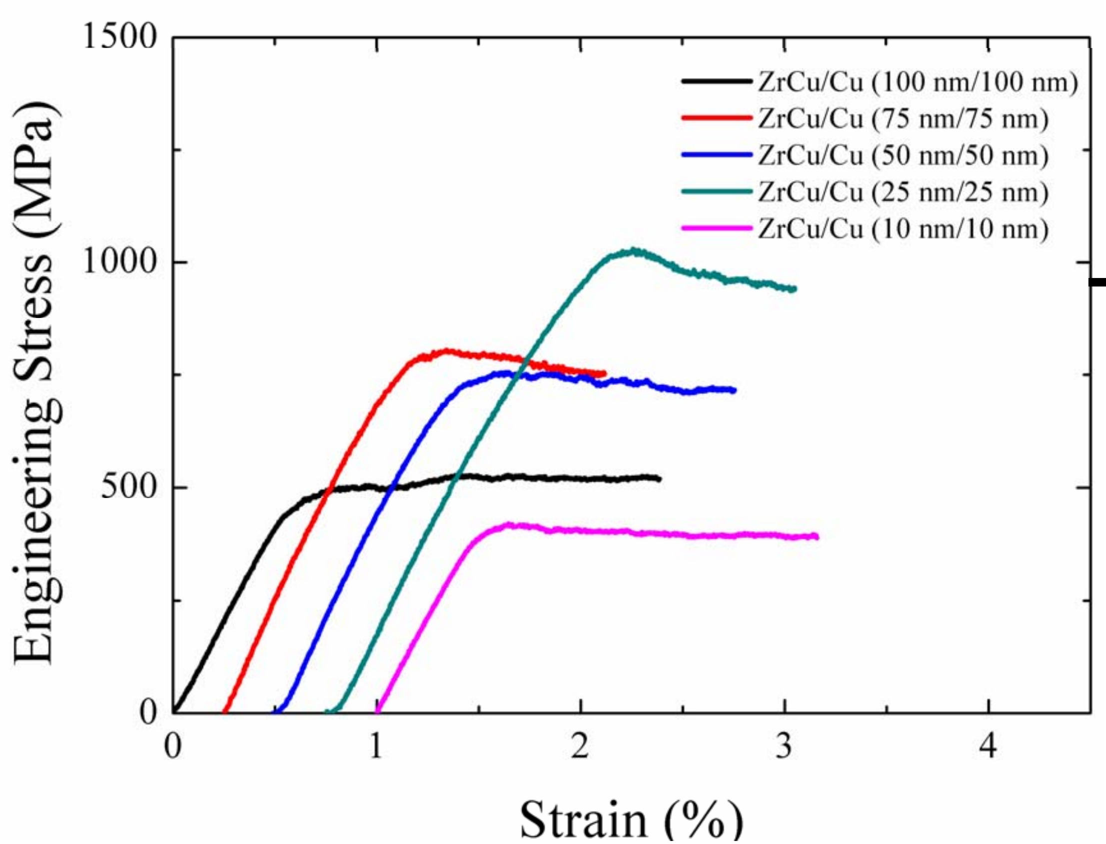


S. G. Mayr and K. Samwer, Phys. Rev. Lett. (2001)



B. W. Sheldon et al. Acta Mater. (2007)

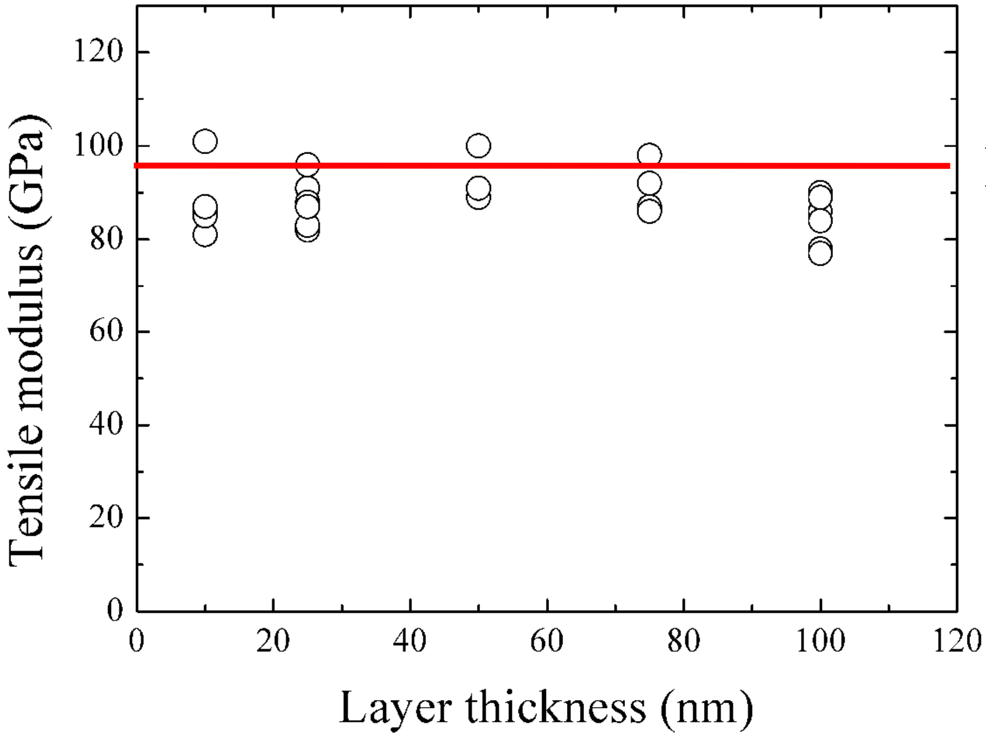
Tensile properties of multilayered TFMGs



Layer thickness (nm)	Tensile modulus (GPa)	Maximum stress (MPa)
100	85 ± 3	550 ± 20
75	88 ± 7	765 ± 30
50	95 ± 5	740 ± 12
25	90 ± 6	1030 ± 15
10	83 ± 6	540 ± 10

The representative stress-strain curves of multilayered thin films. The tensile tests stopped at the strain of ~2%.

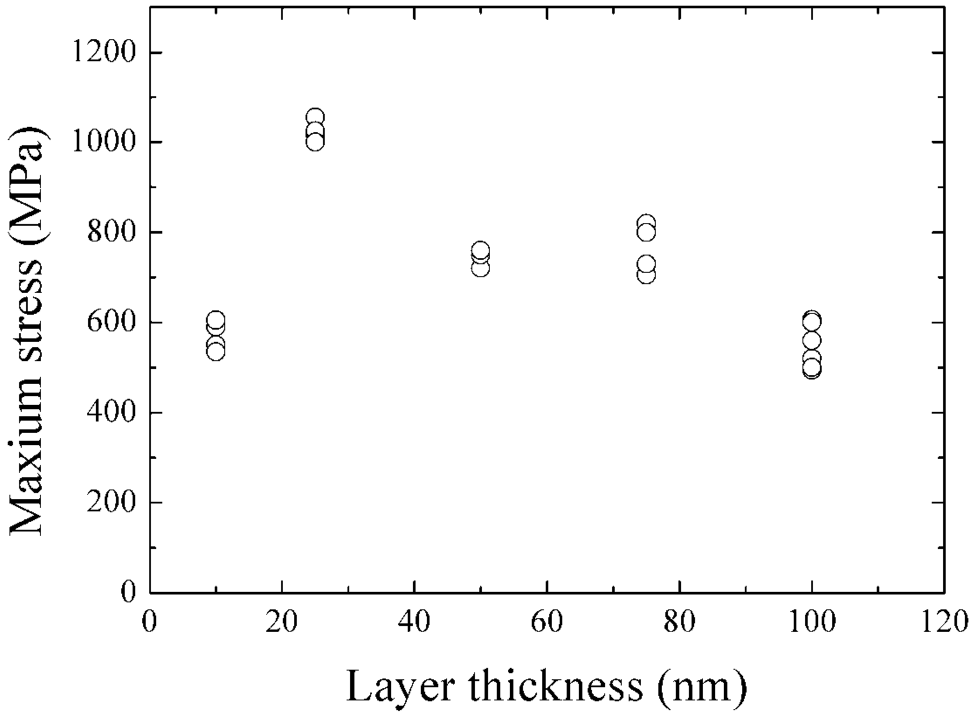
Tensile modulus of multilayered TFMGs



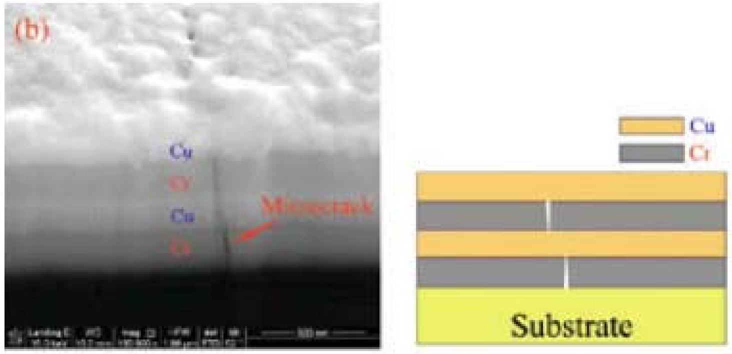
ROM theoretical value

The influence of the layer thickness on modulus is not very significant.

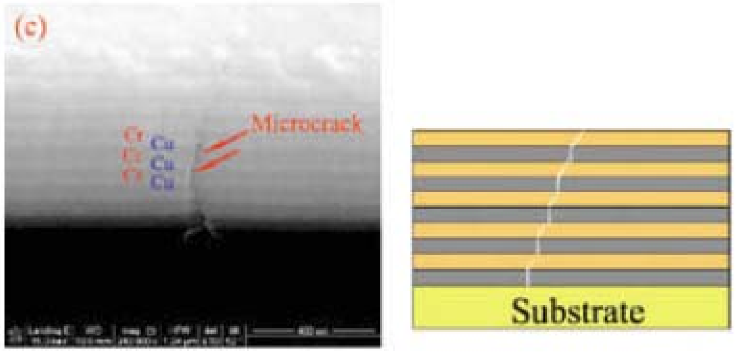
Maximum stresses of multilayered TFMGs



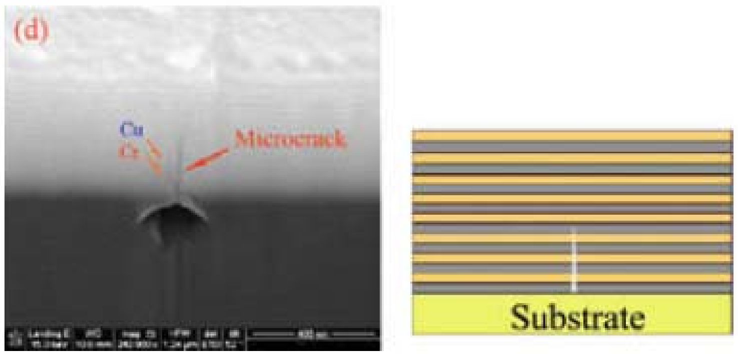
Cu individual layer thickness



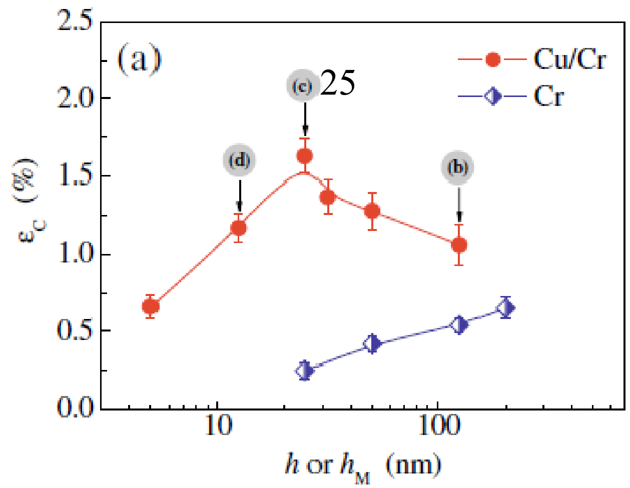
Cu/Cr = 125 nm/125 nm



Cu/Cr = 25 nm/25 nm



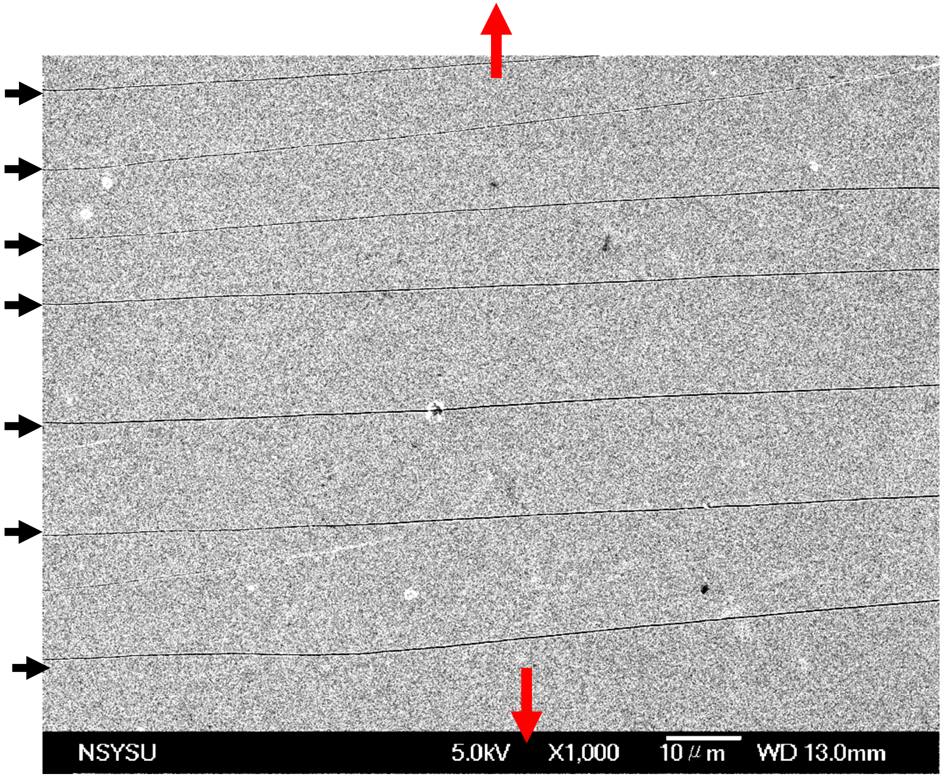
Cu/Cr = 12.5 nm/12.5 nm



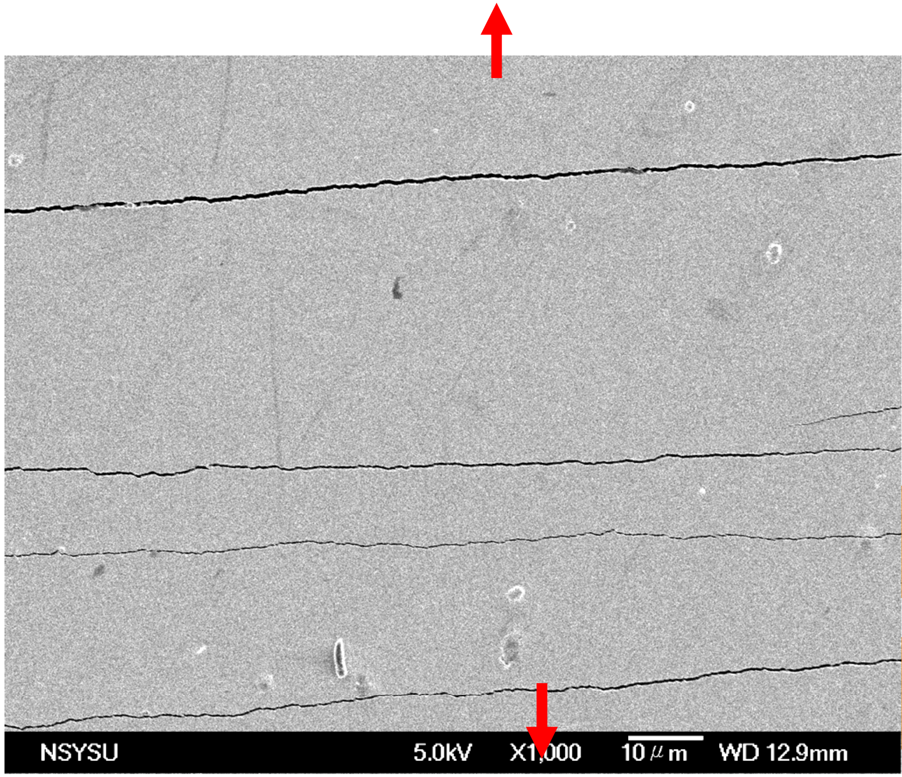
The very thin Cu layers themselves become more and more brittle as weakening the shielding effect of the Cu layers.

J. Y. Zhang et al. Scripta Mater. (2009)

Morphologies of deformed samples

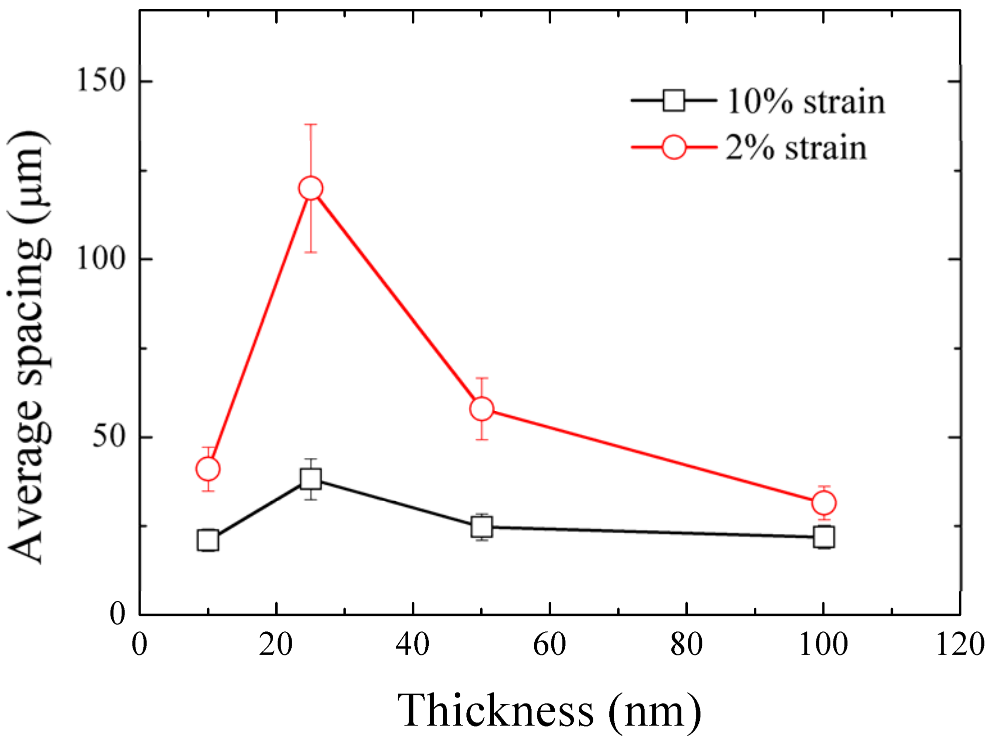


1-μm-thick monolithic amorphous ZrCu



1-μm-thick monolithic amorphous Cu

Averaged spacing between cracks



Conclusions

- The morphology of as-deposited multilayered of ZrCu/Cu thin film is composed of sphere domains, and between the domains there would be stress concentration. The cracks perpendicular to the loading direction would propagate along the domain interface.

- The tensile moduli of monolithic amorphous ZrCu and crystalline Cu thin films are closed to results from micro-compression (93 and 127 GPa).

- The stress-strain curves of the multilayered ZrCu/Cu thin film, extracted by deducting the curves of the uncoated PI substrate from the coated samples are compatible with the theoretically rule of mixture prediction.

- As the layer thickness going down from 100 nm to 10 nm, the tensile moduli would not change too much. The current study demonstrates that the nanolaminate of ZrCu/Cu (25/25 nm) can reach the highest maximum stress (1030 MPa) among all samples.

Thanks

for

Listening