Journal of Zhejiang University-SCIENCE A (Applied Physics & Engineering)

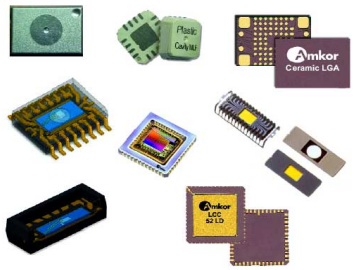
# Experimental and theoretical study on failure behavior of coating-substrate by using SEM *in-situ* method

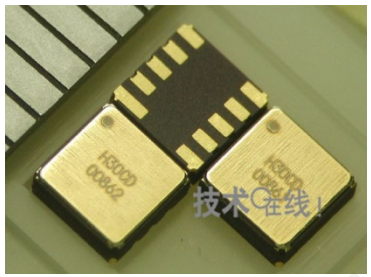
Xi-Shu WANG

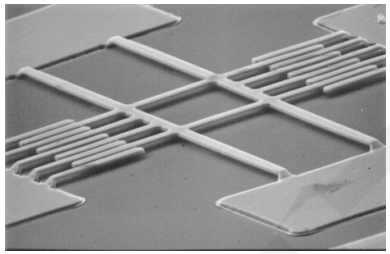
Cite this as: Xi-shu Wang, Xing-wu Guo, Yuzo Nakamura, Hui-hui Yang, Pan Pan, 2016. Evaluation of the critical stress of anodized coating/AZ91D substrate using SEM *in-situ* technology. *Journal of Zhejiang University-SCIENCE A (Applied Physics & Engineering)*, 17(1): 65-75. [doi:10.1631/jzus.A1500178]

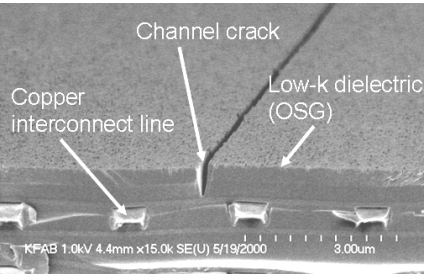
# Background

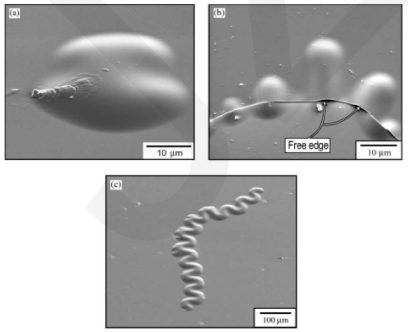
The widely applications of the MEMS and the Reliability issues…





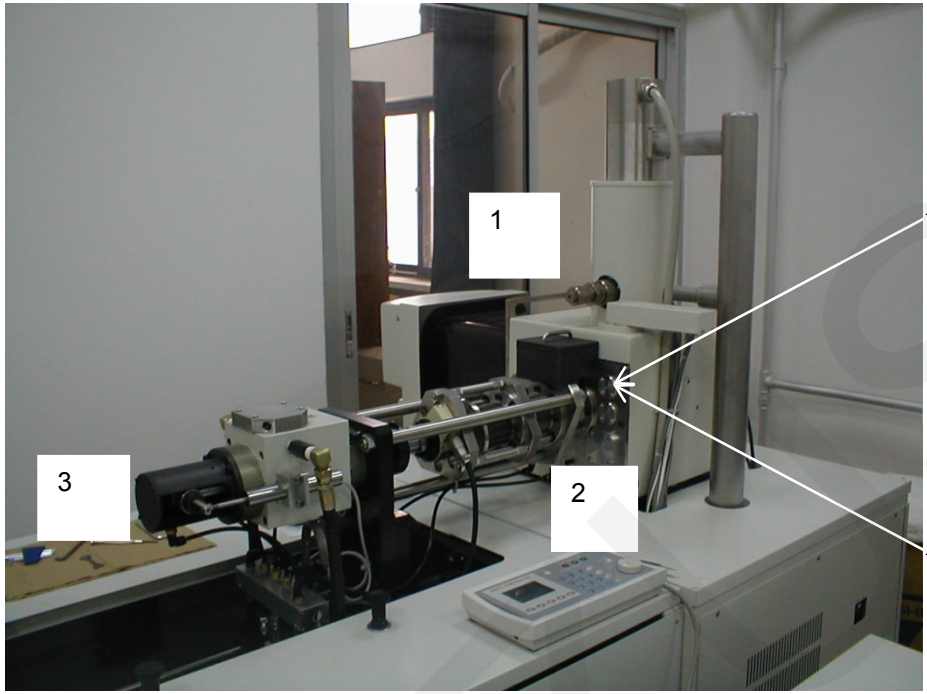

**Critical strain:**

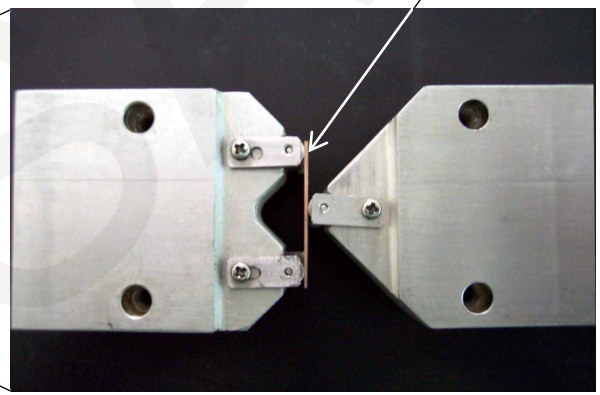
$$\varepsilon_c = \frac{\pi^2}{12(1+\nu)}\left(\frac{h}{L}\right)^2$$

- **Buckling relaxes compressive stress**
- **Bending energy minimizes at long wavelength**

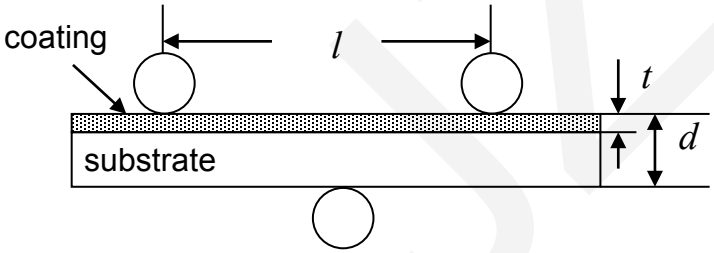
**It is rarely reported that effects of complex stress state and anisotropy on the failure mechanism of coating/substrate structure, especially refers the SEM *in-situ* measure and characterization in meso-scale.**

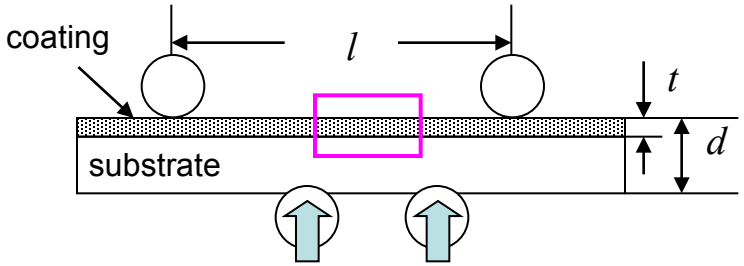
# Experimental method based on SEM *in-situ* technology



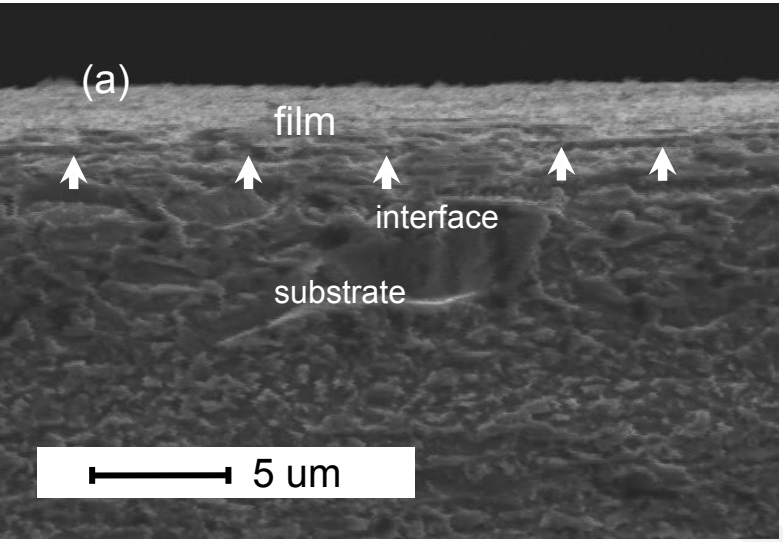

Loading apparatus

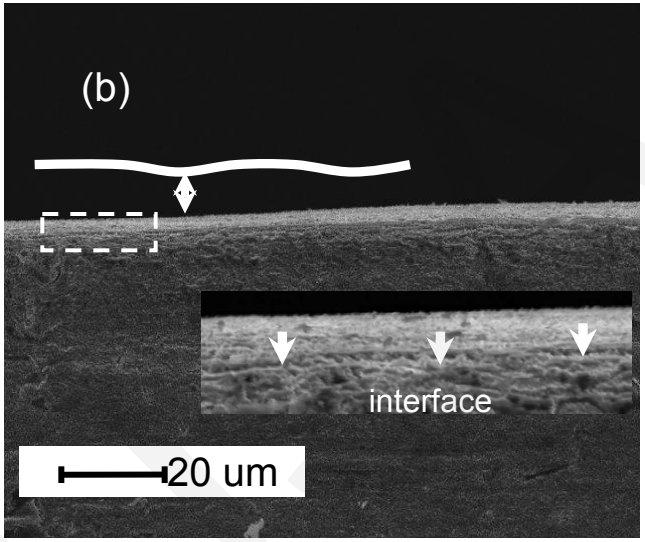


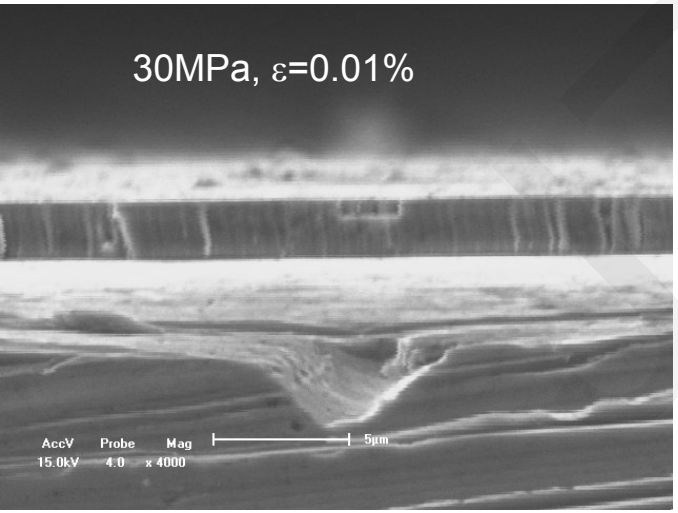



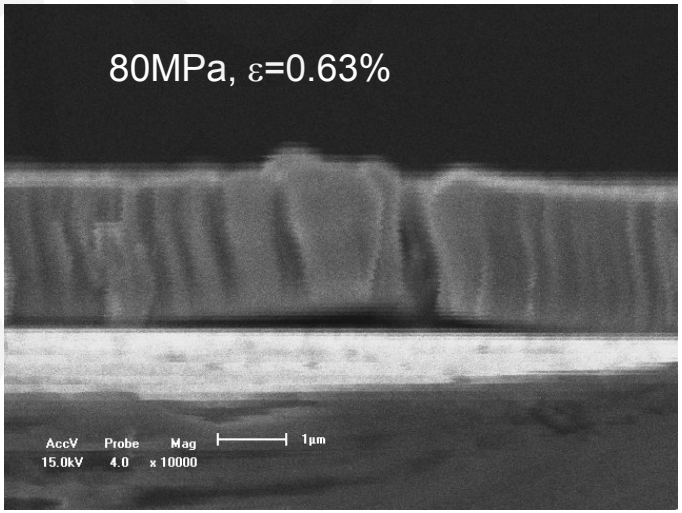

Three point loading and Four point loading

# Results and discussion



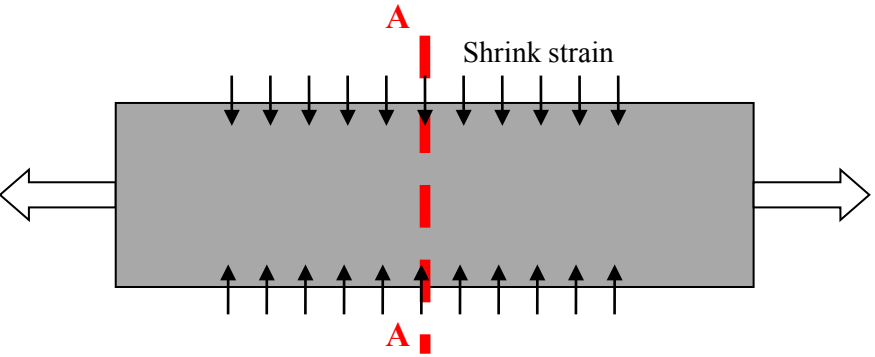

Effect of coating thickness on failure mode of coating-substrate structure can not be ignored under three-point bending loading. There is a critical thickness when the failure mode occurs in conversion process. Seen the authors' relative references as following as:

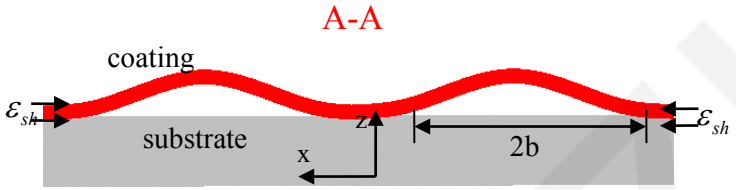
[1] SEM *in situ* study on deformation under three-point bending loading in Cu and Cu/Ni films. *Materials Transactions* 2007; 48(10): 2795-2798.

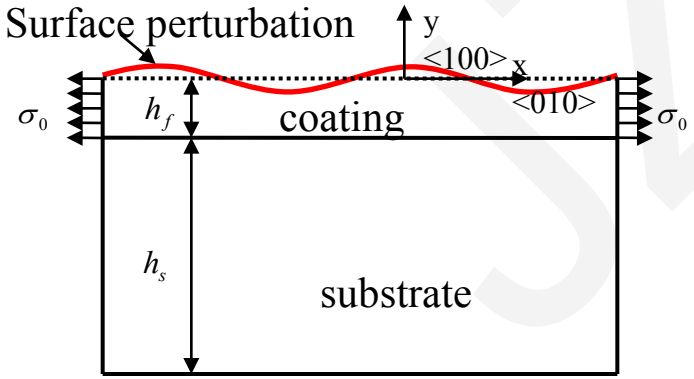
[2] Buckling behavior of metal film/substrate structure under pure bending. *Applied Physics Letters* 2008; 92(13), 131902

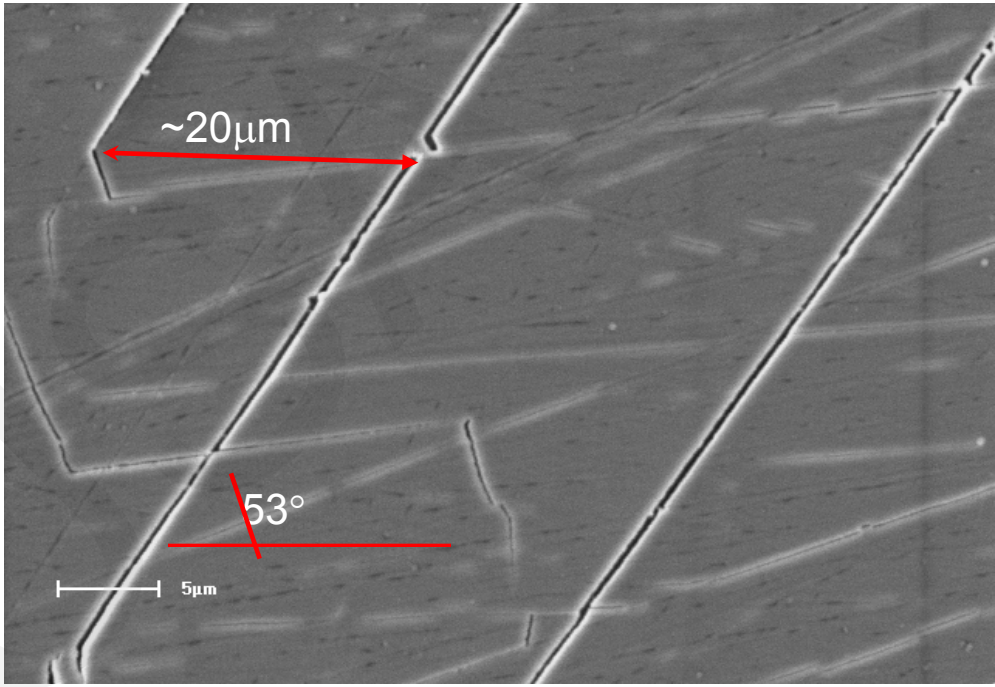
[3] SEM in-situ study on failure of nanocrystal metallic thin films and substrate structure under three point bending. *International Journal of Fracture* 2008; 151:269-279.

# Results and discussion



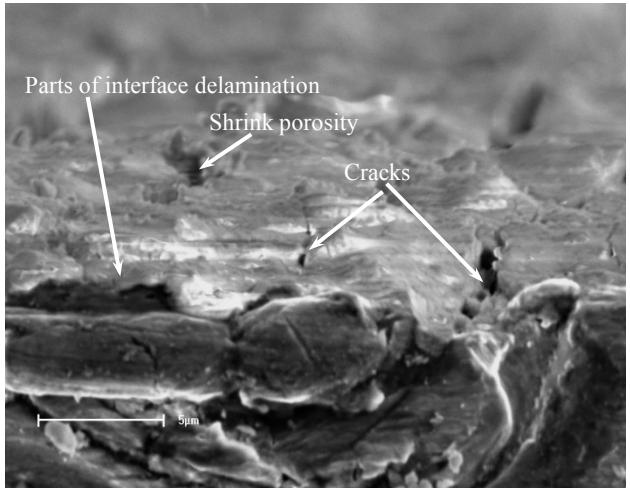



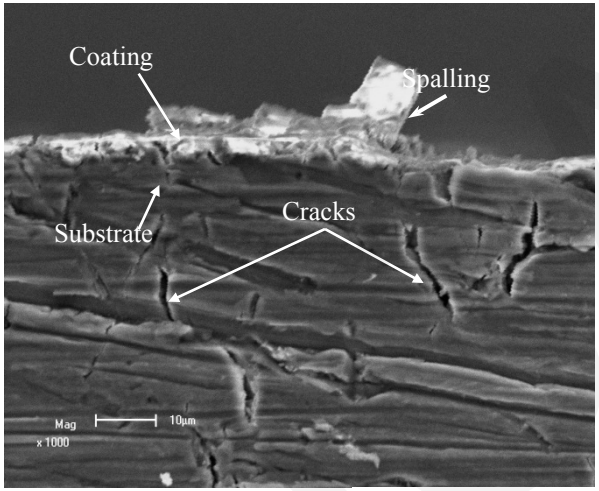



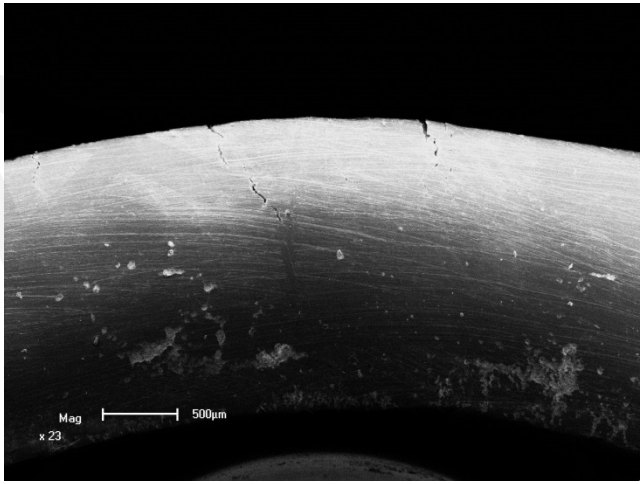



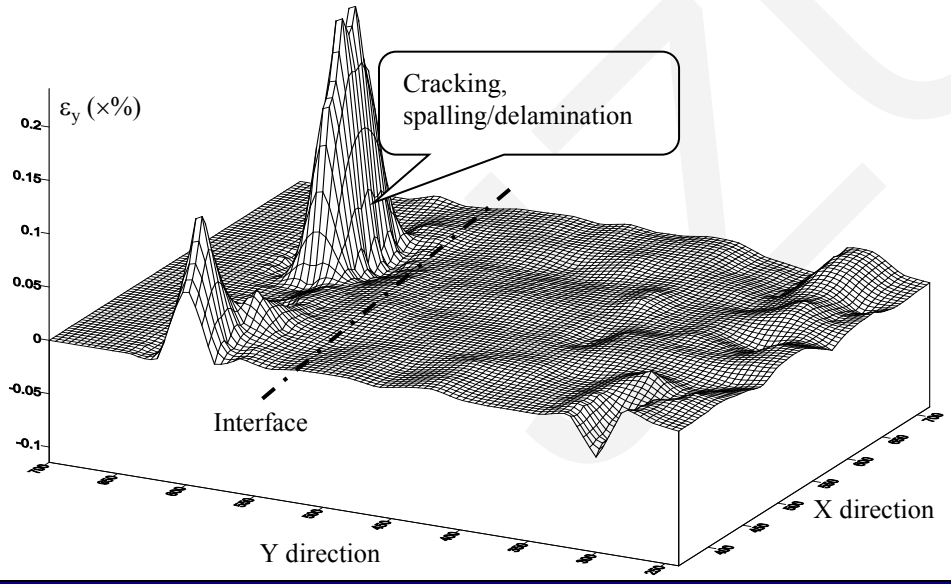

Effects of elastic anisotropic on the surface stability of the film/substrate system. *International Journal of Engineering Science* 2008, 46:1325-1333.

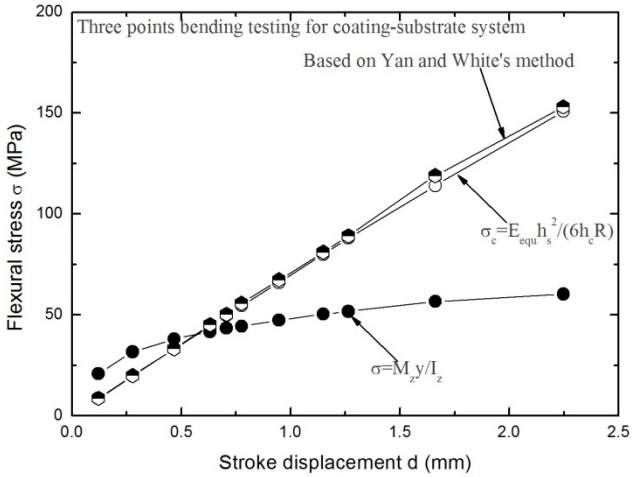
# Results and discussion




For the same coating-substrate structure, there are difference of different characterization methods.

# Conclusions

1) It is important factor for the elastic anisotropic effect on the cracking behavior of thin coating/substrate structure. If the thickness of thin is less than 1 micrometer or K<1，the anisotropic effect could weaken the surface stability so that the thin coating will occur at multi-cracks.
2) If K<1，the critical stress can be expressed by the critical wave length $\lambda_c$：

$$\lambda_c = \frac{8G\gamma\pi}{(k+1)\sigma^2} \text{ or } \sigma = \sqrt{\frac{8G\gamma\pi}{(k+1)\lambda_c}}$$

k=3-4μ (In plain strain state), k=(3-μ)/(1+μ) (In plane stress state).

3) SEM *in-situ* observation cracking direction is about 55 ° tilted to the applied loading direction. It agrees good with the bifurcation theory results. Therefore, the cracking direction can be predicted by the bifurcation theory.
4) SEM *in-situ* observation critical cycling wavelength is about 20 micrometers for 1 micrometer thickness Cu coating/substrate structure under bending loading. It agrees good with the 21 micrometers by the perturbed theory. Therefore, the cracking stress can be estimated by the perturbed theory.
5) When the thickness of coating is over than 2 micrometers for coating/substrate structure, the failure model of coating/substrate structure is different from the thin coating/substrate structure. It is dependent on the interface strength between the coating and substrate similar to the general interface cracking behavior to lead to the coating fracture based on the SEM *in-situ* observation results.
6) The types of failure of an anodized coating-substrate beam under flexural stress are multiple cracks at the coating layer, interface or substrate. Cracks at the interface and in the coating layer are formed preferentially. However, as the flexural stress increases, multiple cracks are initiated and propagated in the substrate. These are accompanied by spalling/delamination of the coating layer in the vicinity of the maximum flexural stress point when the flexural stress is more than 88 MPa, which is lower than the yield strength of an anodized coating-AZ91D substrate structure at 350 V.

# Author's relative references list


[1] Jian-Guo Huang, **Xi-Shu Wang**, Xiang-Kang Meng. SEM *in-situ* study on deformation under three-point bending loading in Cu and Cu/Ni films. *Materials Transactions* 2007; 48(10): 2795-2798.

[2] Li Ying, **Wang Xi-Shu***, Meng Xiang-Kang. Buckling behavior of metal film/substrate structure under pure bending. *Applied Physics Letters* 2008; 92(13), 131902

[3] **Wang Xi-Shu**, Yan Cheng-Kun, Li Ying, Xue Yi-bin, Meng Xiang-Kang, Wu Bi-Sheng. SEM *in-situ* study on failure of nanocrystal metallic thin films and substrate structure under three point bending. *International Journal of Fracture* 2008; 151:269-279.

[4] Li Ying**, Wang Xi-Shu ***, Fan Qin-Shan. Effects of elastic anisotropic on the surface stability of the film/substrate system. *International Journal of Engineering Science*, 2008; 46: 1325-1333.

[5] **Wang Xi-Shu***, Li Ying, Meng Xiang-Kang. An estimation method on failure stress of micro thickness Cu film-substrate structure. *Science China-Technological Sciences* 2009; 52(8): 2210-2215.

[6] **Wang Xi-Shu** Tang Hua-Ping, Li Xu-Dong, Hua Xin. Investigations on the Mechanical Properties of Conducting Polymer Coating-Substrate Structures and Their Influencing Factors. A review. *International Journal of Molecular Sciences* 2009;10: 5257-5284.

[7] **Wang Xi-Shu ***, Guo Xing-Wu, Li Xu-Dong, Ge Dong-Yun. Improvement on the fatigue performance of 2024-T4 alloy by synergistic coating technology. *Materials* 2014; 7: 3533-3546.

[8] **Wang Xi-Shu** *, Guo Xing-Wu, Nakamura Y., Yang Hui-Hui, Pan Pan. Evaluation of the crotical stress of anodized coating/AZ91D substrate using SEM *in-situ* technology. *J. Zhejiang Univ Sci A*, 2015;