EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: Elasto-plastic behaviour of nanometric metallic multilayers	**Experiment number**: SI-1476
Beamline: BM 02	**Date of experiment**: from: 20/06 to: 26/06/2007	**Date of report**: 29/02/2008
Shifts: 18	**Local contact(s)**: Nathalie BOUDET	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

GEANDIER Guillaume*
GIRAULT Baptiste *
RENAULT Pierre-Olivier*
GOUDEAU Philippe
LE BOURHIS Eric
VALAT-VILLAIN Pascale

PhyMat ex-LMP (Laboratoire de Métallurgie Physique) UMR 6630 CNRS/Université de Poitiers
SP2MI, BP 30179, 86962 Futuroscope Chasseneuil CEDEX

Report:

The aim of these experiments was to study size effect on the mechanical behaviour of W layers in W/Cu multilayer presenting discontinued Cu layers when thickness is reduced down to a few nanometres. Specimens were a series of three W/Cu periodic dispersoid composites with a period Λ ranging from 11 down to 1.7 nm and a constant Cu average thickness of 0.2 nm: W10.8/Cu0.2 nm, W2.8/Cu0.2 nm, and W1.5/Cu0.2 nm. These metallic dispersoid composites were deposited by ion beam sputtering on 125 µm thick Kapton® dogbone substrates for tensile tests with a total film thickness of about 200 nm. A W/Cu multilayer realised by magnetron sputtering and composed with 6 nm thick W layers and 18 nm thick Cu layers was studied to go into detail mechanical coupling between W and Cu layers.

We performed *in situ* tensile tests thanks to a dedicated Deben™ deformation device mounted on the 7C diffractometer at BM02 beam line, with a linear detector and X-ray energies of 9.8 or 8.95 keV according to Cu energetic threshold and crystallographic planes targeted. We scheduled the 18 allocated shifts as following:

- 2 for energy and focalisation adjustment, and preliminary tests on our samples;
- 10 for measurements on the W/Cu composite specimen (constant 0.2 nm Cu thickness series);
- 6 for measurements on the W6/Cu18 multilayer.

15 increasing loads were applied to each sample of the dispersoid composite series and 27 different loads to W/Cu multilayer. The $\theta/2\theta$ X-ray measurements were realised with several ψ orientations and two φ values (0 and 90°) for each loading level. The ψ values were chosen according to preferred crystallographic orientations set in the different layers of each sample, that is <110> and <111> fibre-textured for W layers of dispersoid composites, and respectively <110> and <111> fibre-textured for W and Cu layers of W6/Cu18

multilayer. Improved grips have been used to guarantee specimen flatness during loading which greatly reduced uncertainties.

The whole load range was supposed to be included in the sample elastic strain domain (according to prior laboratory tests) except for the greatest period multilayer. In latter case, we could not perform a systematic study and check that the strain remained purely elastic by performing X-ray measurements while unloading the sample. It should be noted that these measurements would not have been possible with a conventional X-ray source, even with very long acquisition times, since uncertainties are of the same order as the peak shifts; here the X-ray synchrotron source allowed us to perform good quality measurements, with a rather high dynamic, in a quite short recording time (around 2 for each loading step).

Preliminary results concern W(310) diffraction peak (for $\psi = 63.4°$ and $\varphi = 0°$) of the W/Cu periodic dispersoid composite presenting the thinnest W layer thickness and the analysis of the W(310) and Cu(222) diffraction peaks (for $\psi = 60°$ and $\varphi = 0°$) of the W6/Cu18 multilayer. Other measurements analysis are currently in progress and must be correlated to transmission electron microscopy, also underway.

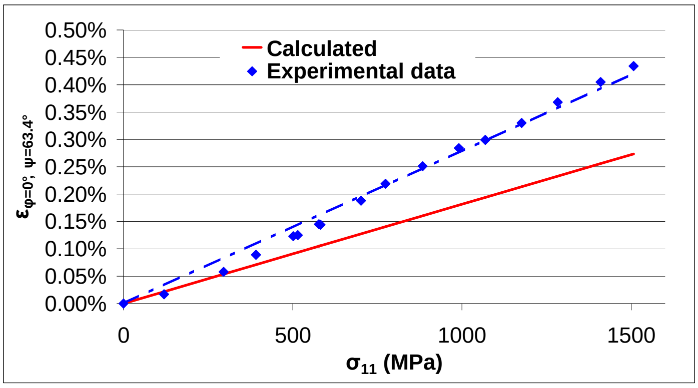

Figure 1: Strain expected considering bulk elastic constants and strain deduced from the shift of the W(310) diffraction peak measurements as a function of estimated applied stress in W layers along the loading axe, for $\psi = 63.4°$ and $\varphi = 0°$, for the thinnest W layer thickness. ($\lambda_{XR} = 0.127$ nm).

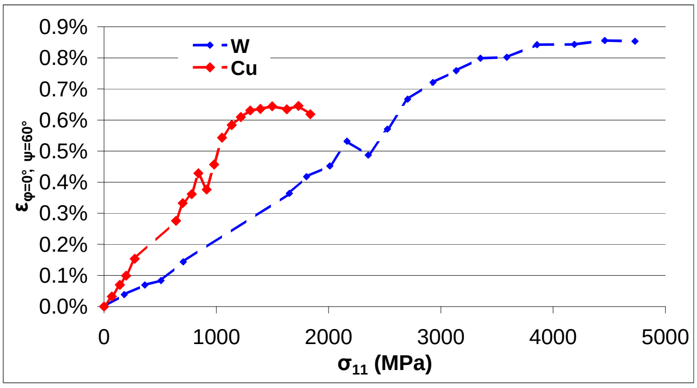

Figure 2: Strain measurements deduced from W(310) and Cu(222) diffraction peak shift as a function of the estimated applied stress in W layers along the loading axe, for $\psi = 60°$ and $\varphi = 0°$, for the W6/Cu18 multilayer. Plastic domain is reached. ($\lambda_{XR} = 0.1387$ nm).

Measurements allow determining strain associated to each loading increment (first loading state taken as a reference). Considering figure 1, XRD-strain in W at $\psi = 63.4°$ as a function of estimated applied stress along loading axis, we can already extract a size effect since linear regression slope is different from the one calculated with bulk elastic constants. The (ε, σ) slope value is much more important than those expected for bulk materials. An elastic softening of W layers and a possible interface roughness even mixing effect may contribute to this variation.

Figure 2 is presenting strain for $\psi = 63.4°$ in both W and Cu layers as a function of estimated applied stress along loading axis. The curves parts present linear behaviours with an available elastic range much more important than those met in former experiments (about 0.1% in W layers). This effect is thought to result from the sample elaboration process. It is of great interest since the main difficulty to determine elastic properties is the intrinsic small deformation measurements.

Conclusions:

Preliminary data analysis shows promising results in terms of size effects on the mechanical behaviour of W/Cu dispersoid composites. Indeed, figures 1 indicates a softening of W layers for the thinner period sample. This softening needs to be confirmed and quantified by means of a thorough analysis of all experimental data (other diffracting plane families). It should be emphasised that strain measurements on the W1.5/Cu0.2 sample would have been extremely difficult and size effect could have been ignored without a synchrotron X-ray source. Efforts have been realised to improve size effect measurements both on deformation device and sample elaboration, showing promising results.

Concerning further experiments, more beam-time would be needed to study plastic yield and deformation thanks to diffraction peak broadening.

These results will be presented at the 2008 fall meeting MRS conference (Oral presentation) and also will be part of the PhD thesis of Baptiste Girault (academic defense: July 2008)