EUROPEAN SYNCHROTRON RADIATION FACILITY

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Application for beam time at ESRF

*(please read the attached guidelines before submitting the completed application form and **20 reduced** copies to the address above)*

Experiment title:	Proposal number *(to be completed by ESRF)*
Microtomography analysis of microvoids in metals. Correlation with ductile fracture mechanisms. **Keywords** (up to 4) : porosity, damage, microtomography	

Please indicate the research area of your proposal:

[ ] Chemistry [ ] Life sciences [ ] Methods and instrumentation
[ ] Soft condensed matter [ ] Hard condensed matter: structures
[ ] Surfaces and interfaces [ ] Hard condensed matter: electronic and magnetic properties
[X] Materials Engineering and Environmental Matters Other:

This proposal is:

[ ] A new proposal [X] A resubmission of : proposal March 00 [ ] A continuation of:
[ ] A proposal relating to research in collaboration with an industrial group

Attachments

You should enclose an experimental report (using our form) on your latest measurements at the ESRF.
[ ] Report on Experiment number(s) attached.
[ ] Report on Experiment number(s) ***relevant to this new proposal*** attached.

Proposer *(to whom correspondence will be addressed)*	
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Beamline(s) requested (give ID or BM number & priorities)	**Number of shifts required** (1shift is 8 hours):	*Preferred starting time:*
ID19 and ID15	10	1. Jan/Feb ___ 2. Mar/Apr ___ 3. May/Jun __ 4. Jul/Aug __ 5. Sep/Oct __ 6.Nov/Dec __ Unacceptable dates: . . . . .

Beam requirements

[ ] 2/3 filling mode [ ] hybrid mode [ ] single bunch [ ] 16 bunch [ ] white beam
[X] monochromatic beam [ ] polychromatic beam Wavelength: . . . . . .

experimental details

Energy/wavelength range: ~ 50 KeV

Beam size on sample: 1.4*1.4 mm^2 (Copper)
0.5*0.5 mm^2 (Tantalum)

Experiment at fixed wavelength: ~0.2 Å

Spot size:

Resolution in energy or momentum:

Other:

Detector system: CCD Camera

sample environment *(supplied by ESRF)*

[ ] Cryostat [ ] Furnace [ ] High pressure [ ] Laser [ ] Closed-cycle refrigerator [ ] Magnet
[ ] Cryogenic gas stream Others:
[ ] Items not supplied by the ESRF:

List all equipment that you will insert into the instrument:

[X] Fixed temperature. Temperature range: 25°

Pressure range:

Indicate requirements for special equipment or facilities:

sample description

Substance **and** formula. Please attach the safety data sheet of the substance. If no safety data sheet is available, please give detailed information about the toxicity of chemical products, and potential fire risks.

. . . . . .

[ ] Powder [ ] Liquid [ ] Gas [X] Polycrystalline [ ] Biocrystallography [ ] Multilayer
[ ] Single crystal Size (in mm^3): 1.4*1.4*4 ≅ 8 (Cu) Mass (in mg): 70 (Cu)
0.5*0.5*4 ≅ 1 (Ta) 17 (Ta)

Surface area: ~ 2 mm^2 (Cu) and ~0.25 mm^2 (Ta) Space group (if known):

Concentration of absorber (ppm or mmol.):

Matrix or solvent:

Unit cell dimensions at T= a = b = c = α = β = γ =

Sample container (capillary, flat plate, pressure cell, etc.)

safety aspects

- If no safety data sheet is included, please give detailed information about the toxicity of chemical products and potential fire risks. Is the sample
  [ ] Radioactive? [ ] A contaminant? [ ] Toxic? [ ] Corrosive? [ ] Explosive?
  [ ] Inflammable? [ ] Oxidizing? [ ] A biological hazard? [X] Without any risk?
- Is there any danger associated with the proposed sample, with any preparation at ESRF, or with sample equipment? [ ] Yes [ ] Uncertain [X] No
  If yes or uncertain, please give details of the risks associated:
- Will you bring live animals onto the ESRF site ? [X] No [ ] Yes; in this case, you need to fill in a special form available from the User Office once your experiment has been accepted.
- After the experiment, will the sample be: [X] Removed by user ? [ ] Stored at ESRF.

I certify that the above details are complete and correct.

Date: Signature of proposer:

To be filled in by ESRF	
Sample environment code	Comments by safety officer

aims of the experiment and scientific background

Description of the fracture of ductile metals [1] relies heavily on void growth models. The approaches which most successfully reproduce macroscopic observations are based on Gurson's model [2], and are in essence effective medium theories.

Assuming a population of isolated voids in an otherwise undamaged matrix, these models establish a link between the temporal evolution of the porosity f (a microscopic information) and the macroscopic plastic deformation rate $\underline{\underline{D}}^p$ via : $\dot{f}(t)/(1-f(t)) = \mathrm{tr}\left(\underline{\underline{D}}^p\right)$ (1)

The goal of this proposal is to investigate the validity of those hypothesis by :

1) Accurately determining the evolution of the porosity by the non-destructive analysis of post mortem damage samples produced with different levels of pressure and check the equation (1).
2) Critically assess the isolated void hypothesis, via a careful analysis of the 3D microtomography images of our samples.
3) Obtaining indications on the void distribution (in size and overlap) which may help to develop more sophisticated models including more realistic distributions.

experimental method

The spall experiment, described in figure (1a) leads to a porosity gradient in the impacted plate. A parallelepiped was cut in each spall plate obtained at different pressures (figure 1b). We have performed a preliminary experiment on ID19 leading to a microtomography image of the shocked sample. We can see on figure(1c) the resulting reconstructed image of a slice in the spalled zone. This image allows to quantify the porosity : number, size, shape, space arrangement, 2D distribution of pores. The next step is, clearly, to perform the same quantification on the 3D image.

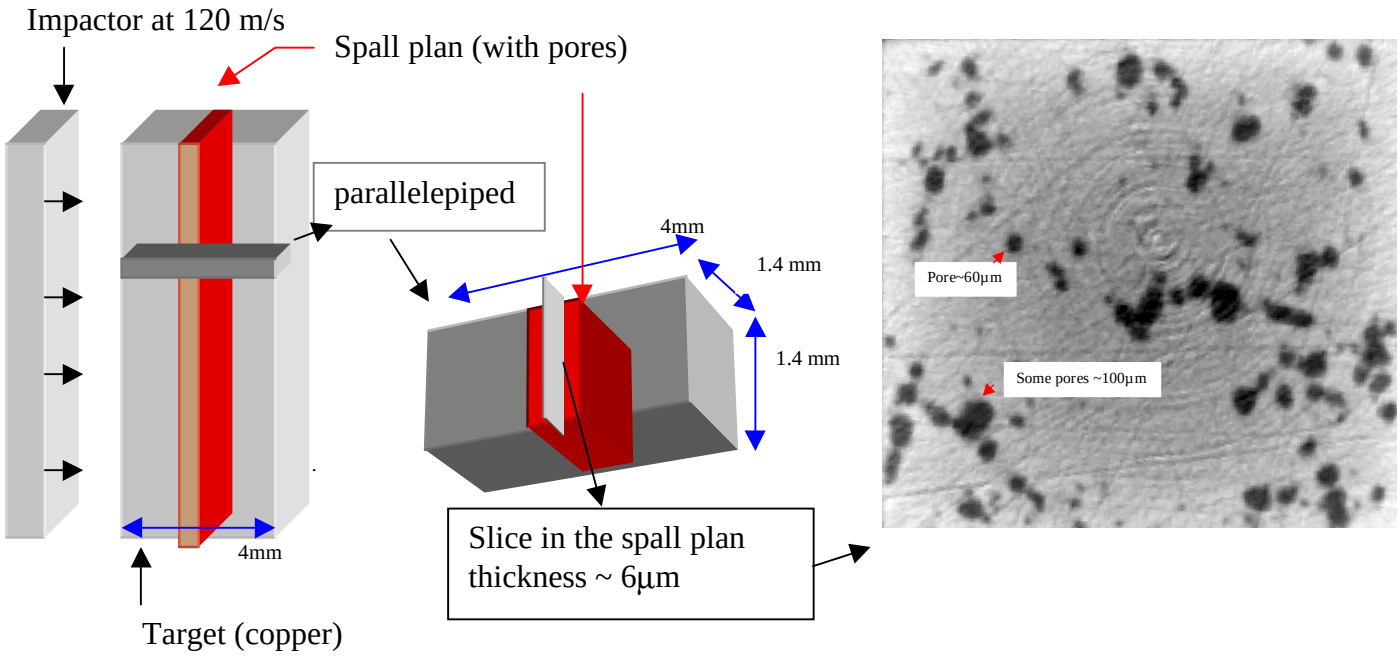

Figure 1(a) : Experiment of spall fracture obtained by plate impact.

(b) Geometry of the cut sample Position of one slice.

(c)Tomography slice of the studied copper FReLoN camera (6 µm pixel size).

results expected

In the preliminary experiment we obtained 400 2D virtual slices of copper (6µm thick), which were analyzed as shown in the preceding slide. We expect to record a series of views, and establish "time dependent" properties.

In the past, metallurgical analysis [3] was the only technique used to get a quantitative result on porosity. The drawback of this technique is clear : it is destructive to metals, especially to copper which is somewhat soft. The cutting, etching and polishing techniques can obstruct and deform voids and lead to inaccurate measurements.

We there fore expect to perform for the first time an accurate 3D assessment of the link between deformation rate and porosity evolution.

References

[1] **Lee Davison, Dennis E. Grady, Mohsen Shahinpoor**
Dynamic Fracture and Fragmentation.
High-Pressure Shock compression of Solids II (1995).
[2] **A. L. Gurson**
Continuum theory of ductile rupture by void nucleation and growth
J.Eng. Mater. Technol., Trans. Of ASME 99, 2 (1977).
[3] **A.K. Zurek, M. A. Meyers**
Microstructural Aspects of Dynamic Failure.
High-Pressure Shock compression of Solids II (1995) pp25-68.

What are the technical reasons which make ESRF necessary for your experiment ? why are other synchrotron radiation sources not appropriate ?

The spatial resolution of both ID19 (1µm) and ID15's (5µm) tomography is very precise. With this technique it is possible to see a complete range of pore size (a few microns to one hundred microns). It is a non destructive technique.
The 3D reconstruction of porosity for samples is possible.

Have you previously done an experiment using synchrotron radiation ?

[X] Yes, at . ESRF. . . . . [ ] No.

Have you already used synchrotron radiation for ***this*** project ? [X] Yes [ ] No.
Have you used synchrotron radiation at the ESRF ? [X] Yes [ ] No.

Publications

Please note below the references of all papers published during the past 18 months as a result of measurements which you have done at the ESRF. (If space is insufficient, please attach a list.)