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Evaluation of a disposable type of 6–6 frame for high-pressure experiments

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A great success of developing a unique kit that facilitates assembling second-stage anvils for a multianvil press using in a 6─6 mode has made us launch a new project of developing a disposal frame for the assembly. We tested an acrylic frame in FY2020.

1 Introduction

A compression method using anvils in a tandem fashion for a multianvil press has become popular in high-pressure science and engineering. A plastic frame has led to a revolution in preparing a second-stage assembly when operating in a 6–6 mode; a plastic frame designed by the present authors with an assembling kit has made it possible to set second-stage anvils within a few minutes without loss of precision required to attain high pressure [1]. The efficiency exceeds more than ten times compared to the standard approach using a metallic frame.

However, in only one point, a plastic frame is less efficient compared to a metallic frame in that it is no longer reusable after it experiences a blowout, even if no visual damage such as breakage or deformation is recognizable. In contrast, a metallic frame, which can absorb a shock by disassembling into the parts, is usually reusable. The authors manufactured a plastic frame made of PEEK or POM, which is by itself rather expensive. In some cutting-edge science and technology, researchers often conduct high-pressure experiments until a blowout occurs.

Thus, the authors have started a new research and development project to produce a disposable plastic frame for those researchers.

2 Candidate plastic materials

The project started with searching candidate plastic materials with high melting points and machinability. Nevertheless, at the same time, they must inexpensive compared to the cost of PEEK or POM. After some machining tests for various candidate plastic materials, acrylic and nylon plastics were finally shortlisted. In FY2020, we have examined an acrylic frame first. However, a minimal period of beamtime due to the COVID-19 calamity has made the examination incomplete.

3 Experiment

Because a load of more than a hundred tons may be applied in high-pressure experiments, we must carefully align and adjust both the first- and second-stage anvils in a 6–6 operation; those anvils must share the ideal center within a press's high-pressure vessel. More precisely, we have to align the center of second-stage anvils to the first-stage (outer) ones already mounted on a press. We call this alignment of the second-stage anvils centering. The assembly kit developed by us ensures the centering with very high precision on assembling the second-stage anvils. However, we need to confirm *in situ* that the centering remains ensured on applying a load.

We conducted in situ confirmation of the centering condition using MAX-80 installed at NE5C. We set a W–Re wire piece of diameter 0.125 mm and length 1 mm, used as a dummy sample, in a high-pressure cell made of boron and epoxy resin. We placed the dummy sample to come to the common center of the anvils when the centering condition was satisfied. We then confirmed the centering aspect by transmitted x-ray images. A typical compression and decompression cycle were as follows: We first applied 30 tons of load at room temperature and then heated a dummy sample to 1000 K. We further loaded up to 100 tons at maximum at that temperature. We continually monitored the frame temperature during a cycle. We measured pressure at the sample position using a pressure standard, NaCl, at every addition of 20 tons of load. We also took a photo of the transmission image of the dummy sample at those pressures.

The COVID-19 catastrophe severely limited the experimental opportunities; we could have only one beamtime in FY2020. We have planned to test TEL of 4, 6, and 8 mm as the second-stage anvils. However, a testing TEL of 4 mm anvils remained unfinished.

3 Results and Discussion

During keeping the sample at 1000 K under pressure, the frame's temperature did not far exceed 40 degrees centigrade, as experienced for PEEK and POM frames. The frame's highest temperature was still below half of the nominal melting temperature, 100 degrees centigrade, of an acrylic frame. Therefore, as for the temperature environment, no problems may happen for using an acrylic frame to bundle the second-stage anvils.

A CCD camera placed just behind the sample on the downstream side of the beam made it possible to observe the achievement of centering by monitoring transmitted images. As an example, we show in Fig. 1 a situation we observed when we compressed the sample using a set of secondary anvils with a truncated edge length (TEL) of 6 mm. The anvils have a ditch of 1.1 mm width and 0.4 mm depth on their face to secure a relatively wide anvil gap upon compression. Recall that the assembly kit placed a dummy sample at the center (Fig. 1(a)). The sample was always sitting at the middle of the anvil gap even under the highest load of 100 tons applied (Fig.1(d)).

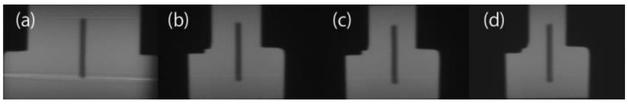

Fig. 1: A transmission image of the dummy sample (a W–Re wire of 0.125 mm diameter and about 1 mm length) in an anvil gap when an acrylic frame was employed to bundle the second-stage anvils of TEL 6 mm is depicted. The anvil faces, between which the x-ray beam passes, were ditched to widen the gap. (a) Anvil gap 2.11 mm at ambient conditions, (b) 0.94 mm at 3.1 GPa and room temperature, (c) 0.91 mm at 2.6 GPa and 1000 K, and (d) 0.72 mm at 6.0 GPa and 1000 K.

The high accuracy attained in the centering on the initial alignment was brought about by the assembly kit, which ensures compatibility not only for PEEK and POM frames but also an acrylic frame used in the current examination. The uniform smoothness of the acrylic frame surface, which remains untransformed under loading at a high temperature such as 1000 K, produced the uniform movement of all six anvils supported by the frame, keeping the centering accuracy.

However, it may be difficult for an acrylic frame to raise a sample temperature beyond 1000 K because such an operation may readily bring the frame's temperature beyond half of its melting point. Such usage may pose a safety risk; a PEEK or POM frame should be used instead.

Thus, PEEK and POM frames, which cost more than double, offer a better temperature environment than an acrylic frame. We noticed through actual use an advantage of an acrylic frame, not inherent in PEEK and POM frames; an acrylic frame is transparent. The transparency makes it possible to verify that six anvils are properly configured visually. In particular, we can visually confirm the contact of screws temporarily fixing the anvils to the frame before setting the second-stage assembly at the center of the first-stage anvils. Due to this transparency, an acrylic frame looks beautiful. However, this subsidiary feature will contribute to making a good sale when the frame is released.

4 Conclusions

Up to this point, we can conclude that an acrylic frame has met the requirements to realize high-pressure and high-temperature experiments using a multianvil press in a 6–6 mode. However, unlike PEEK and POM frames, an allowable highest temperature, measured at a sample position, for an acrylic frame may be 1000 K. When one tries to raise a sample temperature beyond 1000 K, we strongly recommend adopting a PEEK or POM frame. The final approval will be subject to verification tests using the second-stage anvils of TEL 4 mm, which will be done in FY2021.

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References

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