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Evaluation of another disposal type of 6–6 frame for high-pressure experiments

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We reported in FY2021 the performance of an acrylic frame, although all the examinations have not been completed yet. Here, we report the result of a nylon frame, the prime candidate for the cost, tested under relatively severe high-pressure conditions.

1 Introduction

We have already stated the purpose of the current project in the last report [1]; to look for low-cost plastic materials and test a frame made of them, supporting the second-stage anvils when used with a multianvil apparatus in a 6–6 compression mode. The material should have similar machinability of cutting and grinding to that of very expensive PEEK or POM. A PEEK or POM frame is never reusable when it experiences a blowout [2]. This situation warrants exploiting a disposable frame.

We have examined a frame made of acrylic materials to reach the goal. However, the COVID-19 pandemic made us abandon completing the whole tests scheduled. In FY2021, we inspected a nylon frame in advance of the completion because nylon materials were the cheapest among the materials we prepared, and therefore a nylon frame should be the prime candidate to meet our requirements.

2 Experiment

The inspection method is fundamentally the same as that described in the previous report [1]. The critical points to be checked are: (i) the accuracy of the initial sample centering and (ii) the stationarity and stability of the sample's centering during compression. Here, the "centering" means adjusting the virtual of the second-stage anvils supported by a frame in question to coincide with that of the first-stage anvils.

The condition (i) will be fulfilled when the assembling tools of the second-stage set developed in the previous study [2] work perfectly. The tools not only ensure accurate centering but also allow everyone to assemble the second-stage set, consisting of a set of anvils and a frame, in a few minutes. (Without using the tools, it will take at least half an hour even for an expert to assemble the second-stage set with comparable accuracy.)

Once condition (i) is satisfied, a constant movement of whole second-stage anvils during compression will fulfill the condition (ii). The anvils displace uniformly unless a plastic frame's surface quality changes while keeping the sample at high temperatures.

A compression and heating cycle consisted of the following processes: First, 40 tons of load was applied to a high-pressure cell holding at the center a W–Re wire segment of a length of 1 mm and a diameter of 0.125 mm as a dummy sample to judge the centering aspect. This initial loading was conducted at room temperature. The sample was then heated up to 1000 K, and the cell was isothermally loaded up to 100 tons. We measured the sample in situ through transmitted x rays along the sample path. The pressure at the sample position was measured using a pressure standard composed of NaCl. Finally, the cell was cooled down to room temperature and was completely unloaded.

We used MAX80 installed in beamline NE5C at AR as a multianvil apparatus. We found in 2020 that the upper surface of the bolster plate of the press was not parallel to the bottom surface of the slide. The inclination turned out to rotate the second-stage set upon compression. The rotation often promoted a blowup when loading beyond dozens of tons. Twice overhauls during FY2021 almost removed the failure. An improvement in the plates' parallelism resulted in a broader anvil gap. However, these overhauls made the project run behind schedule.

3 Results and Discussion

We present here the most recent results for examining a nylon frame. The complete results, including those for an acrylic frame, will be published as a technical paper after the final mission scheduled in the first term of FY2022.

Centering precision

Figure 1 shows x-ray transmission images taken during the last two runs (LS28 and LS29). A dummy sample was initially located at the correct position in both runs, i.e., almost the center of the anvil gap before loading. The assembling tool developed in our previous study brought this correct initial setting of a high-pressure cell.

As compression proceeded, the sample was slightly inclined (probably caused by the very insensible inclination of the horizontal plates installed in MAX80) in L29. However, the sample was still in the middle of the gap even at the highest load applied, implying that the whole second-stage anvils supported by a nylon frame moved uniformly during the sample was kept even at 1000 K.

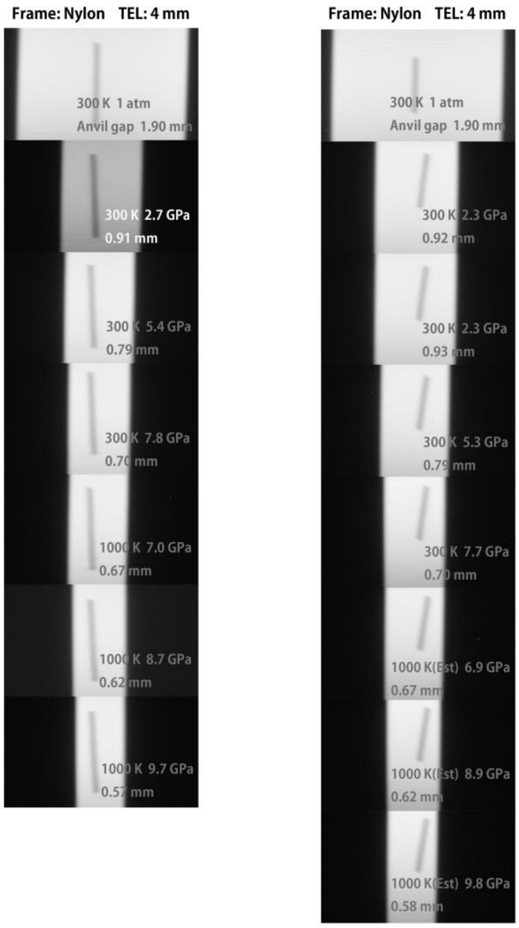

Fig. 1: X-ray transmission images of a dummy sample taken in LS28 (left) and LS29 (right) at the thermodynamic conditions indicated are organized vertically in increasing order of the load applied. We can always recognize the sample at almost the center of an anvil gap, with its width also indicated.

Frame's temperature

The upper panel of Fig. 2 depicts the nylon frame's temperature profiles when the sample followed the pressure–temperature paths shown in the lower panel. Frame's temperature did not exceed 40 degrees centigrade when we used second-stage anvils with a TEL of 6 mm or above. When we used the anvils with a TEL of 4 mm, the nylon frame experienced, for the first time, its temperature exceeding 40 degrees while holding a sample at 1000 K. Our test experiments used a high-pressure cell made of boron and epoxy resin, which conducts heat relatively well. Because a high-pressure cell with its center kept at a high temperature such as 1000 K is held by the anvils, heating will be accelerated when using anvils with a smaller TEL because the distance between the anvils, determined by a side length of a high-pressure cell, decreases with decreasing TEL. Our experiments thus far suggest that a TEL of 6 mm is marginal to keep the frame's temperature not exceeding 40 degrees centigrade (as far as the sample is kept at 1000 K at most). Because a PEEK frame remained at 35 degrees under the same experimental conditions, this temperature rise of a nylon frame is likely ascribed to the thermodynamic properties of nylon itself.

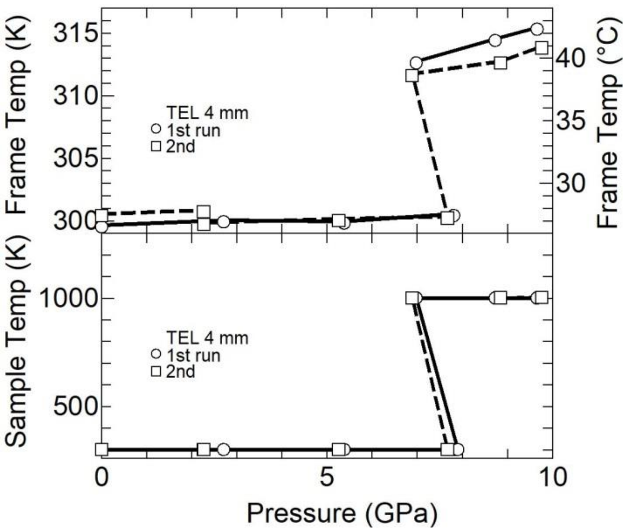

Fig. 2: The bottom panel shows the temperature-pressure paths of LS28 (1st run) and LS29 (2nd run), whereas the top panel shows the variations of the nylon frame's temperature plotted as a function of pressure. The sample temperatures of LS29 were estimated based on the power-temperature relationship.

4 Conclusions

Our experiments demonstrated that nylon could be used as a plastic material alternative to expensive PEEK and POM. From the cost aspect, nylon resin must be a promising material for a disposable frame used for a multianvil apparatus in a 6–6 compression mode. However, unlike a PEEK or POM frame, an allowable upper bound for a sample temperature will be limited to 1000 K for safe experiments.

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References

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