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## APPLIED PHYSICS

## Dynamic fracture of tantalum under extreme tensile stress

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The understanding of fracture phenomena of a material at extremely high strain rates is a key issue for a wide variety of scientific research ranging from applied science and technological developments to fundamental science such as laser-matter interaction and geology. Despite its interest, its study relies on a fine multiscale description, in between the atomic scale and macroscopic processes, so far only achievable by large-scale atomic simulations. Direct ultrafast real-time monitoring of dynamic fracture (spallation) at the atomic lattice scale with picosecond time resolution was beyond the reach of experimental techniques. We show that the coupling between a high-power optical laser pump pulse and a femtosecond x-ray probe pulse generated by an x-ray free electron laser allows detection of the lattice dynamics in a tantalum foil at an ultrahigh strain rate of  $\dot{\epsilon} \sim 2 \times 10^8$  to  $3.5 \times 10^8 \text{ s}^{-1}$ . A maximal density drop of 8 to 10%, associated with the onset of spallation at a spall strength of  $\sim 17 \text{ GPa}$ , was directly measured using x-ray diffraction. The experimental results of density evolution agree well with large-scale atomistic simulations of shock wave propagation and fracture of the sample. Our experimental technique opens a new pathway to the investigation of ultrahigh strain-rate phenomena in materials at the atomic scale, including high-speed crack dynamics and stress-induced solid-solid phase transitions.

## INTRODUCTION

Dynamic high-pressure physics has gained increasing interest during the last decades in various domains such as planetology, including high-pressure phase diagram studies (1), crater impacts (2, 3), and hypervelocity impacts from micrometeoroids and orbital debris (4, 5), as well as technological and advanced material applications (6–9) such as laser shock peening (10), development of new materials (11), or adhesion tests (12). An important phenomenon for these studies is the dynamic fracture of a material under high-speed loading, which is critical for understanding its fundamental mechanical properties under shock loading. Dynamic fracture is characterized by the ejection of one or

multiple fragments because of tensile stresses. The failure process under dynamic compression is due to the crossing of two release waves: In the case of a shocked material plate, one of these rarefaction waves is generated by unloading, starting from the front face of the plate, whereas the second one returns from the rear free surface after the reflection of the shock on it (13, 14). Several experimental techniques have been developed to study the effect of dynamic damage (spallation) on materials at different strain rates and temperatures using macroscopic information, such as the evolution of free surface velocity and/or postmortem examination of the sample (15–17). However, no direct ultrafast observation of the stretching of the lattice at extreme deformation rates has been performed to date. More specifically, the high-pressure fracture properties of metals (such as Ta) or metal alloys are important for many applications, including for micrometeoroid and orbital debris shielding for future spacecraft and new generations of satellites, for plasma-facing components in nuclear power plants (18) and even for laser peening and adhesion tests in a range of industrial applications. Previous experimental works (19–22) have all been restricted to indirect, macroscopic observations of the spallation process. Molecular dynamics (MD) simulations performed at strain rates of  $\dot{\epsilon} > 10^8 \text{ s}^{-1}$  (23) predict that the spall strength of Ta should be extremely high ( $> 10 \text{ GPa}$ ), a pressure that could be calculated from an x-ray diffraction (XRD) signal.

Here, we report experimental results taking advantage of coupling a femtosecond x-ray free electron laser (XFEL) and a high-power optical laser beam to investigate, at the lattice level, the spallation (dynamic fracture) of a 5-μm polycrystalline Ta sample at extreme deformation rates,  $\dot{\epsilon} \sim 2 \times 10^8$  to  $3.5 \times 10^8 \text{ s}^{-1}$ , and spall strength of 16.8 GPa. These results are then directly compared to large-scale MD simulations.

More generally, the study presented here shows an experimental technique, allowing for observation, at the lattice level, of the stretching

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of a material under extreme deformation rates and determination of one of its universal properties: the spall strength. At this scale, it is then possible to directly compare the experimental results with MD simulation to develop and/or further constrain interatomic potential for a given material. This method has strong implications for material characterization and optimization, investigation of material properties under high impact, or the development of new materials as it bridges the gap in the understanding of the relationship between atomic structure and material properties.

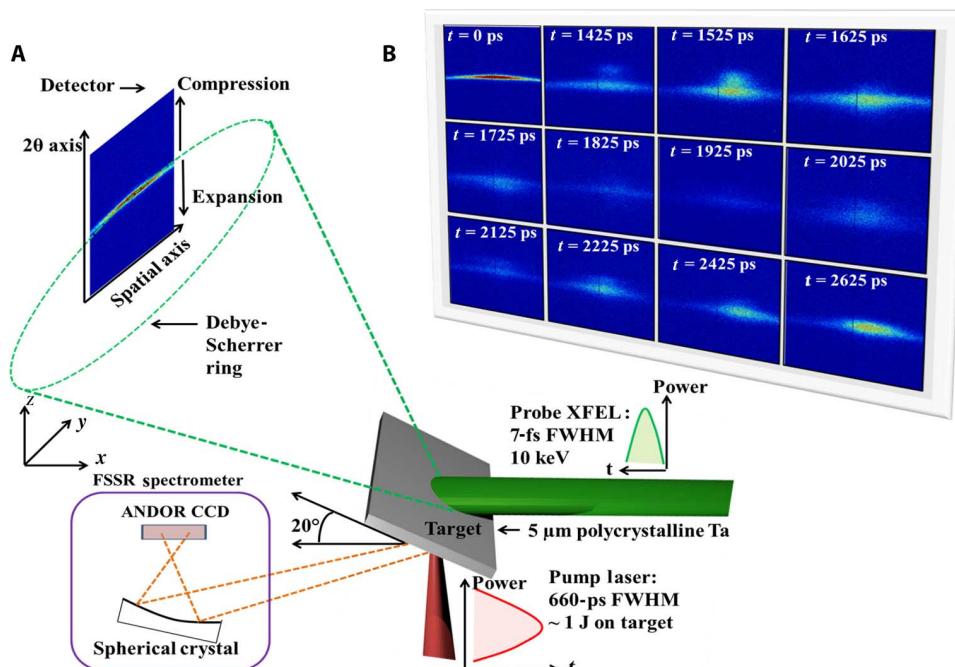
## RESULTS AND DISCUSSION

The pump-probe experiment was performed at the SPring-8 Angstrom Compact Free Electron Laser (SACLA), Japan (24, 25). The experimental setup is shown in Fig. 1. An optical laser at 800-nm wavelength, with a pulse duration of ~660-ps full width at half maximum (FWHM) and an energy on target up to 1 J, was focused down to ~280- $\mu\text{m}$  FWHM, resulting in an on-target intensity of  $\sim 2.5 \times 10^{12} \text{ W/cm}^2$ , to generate a shock wave in a 5- $\mu\text{m}$ -thick polycrystalline Ta target. At the rear side of the target with respect to the pump laser, a 7-fs quasi-monochromatic XFEL pulse, with ~10-keV photon energy (energy bandwidth of ~0.5%) and  $\sim 10^{11}$  photons per pulse, was used to probe the crystallographic lattice spacing  $d$  of the sample. The target was placed at a 20° incident angle compared to the XFEL beam axis, which was focused in one direction down to ~17  $\mu\text{m}$  using a mirror, whereas the other axis was adjusted to ~200  $\mu\text{m}$  by a two-quadrant slit. X-rays were then diffracted by the orderly array of the body-centered cubic (bcc) (002) Ta planes into a Debye-Scherrer diffraction ring. A part of the diffraction ring was observed on a one-megapixel array detector (26) to determine the lattice spacing  $d$  of the sample (see Fig. 1A). In that way, a higher 20° angle would

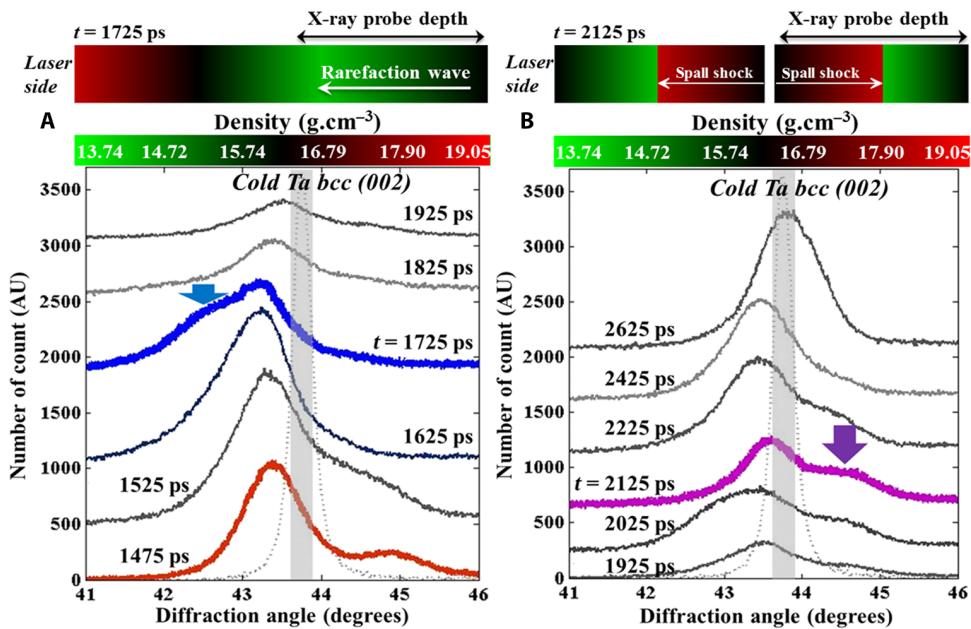
correspond to a compression, whereas a lower 20° angle refers to an expansion of the lattice (see the Supplementary Materials). Note that our experimental configuration allows us to study the lattice deformation and fracture of the Ta sample in a region corresponding to the submicrometer penetration depth of the 10-keV x-rays inside the material ( $L \sim 0.85 \mu\text{m}$ ). This short penetration depth means that our data are only sensitive to a small portion of the sample, rather than integrating over the whole thickness of the sample as in a transmissive configuration (27), and are therefore extremely sensitive to the atomic motion very near the sample free surface. In addition, this configuration separates the probed region from the laser-matter interaction occurring at the front surface of the target, allowing us to probe a region in the target where the shock wave is well established.

The evolution of lattice deformation associated with the ultrafast fracture in Ta is given by a time series of XRD patterns taken by varying the delay between the pump and the probe beam, where  $t = 0$  is defined at the beginning of the laser pulse (see the Supplementary Materials). The mean crystallite dimension ( $\tau$ ) of our sample is on the order of ~20 nm, related to the broadening of the measured peak at zero pressure and room temperature (see Fig. 1B) by the Scherrer equation.

The 20° angle of the unstrained bcc (002) lattice plane of our Ta sample measured in the experiment is 43.76° (see Fig. 2) corresponding to a spacing  $d = 1.652 \text{ \AA}$  and density of  $\sim 16.66 \text{ g/cm}^3$ . Within the first 50 ps presented in Fig. 2 ( $t = 1475$  and 1525 ps), one can observe two or more peaks related to a compression and an expansion (stretching) of the lattice in a part of the x-ray probed zone (see Fig. 2A). The observed compression ( $20 > 43.76^\circ$ ) at these early times corresponds to the shocked compressed part of the sample. The simultaneous expansion ( $20 < 43.76^\circ$ ) is associated with the propagation of the rarefaction wave in the compressed sample that was produced when the shock wave



**Fig. 1. The pump-probe experiment at SACLA.** (A) Experimental configuration, where a 5- $\mu\text{m}$ -thick polycrystalline Ta sample is compressed by a pump (optical) laser and the diffraction is observed preferentially in the bcc  $<001>$  direction. An ultrafast (7 fs) x-ray beam focused in the  $z$  direction probes the lattice arrangement of the sample and generates a Debye-Scherrer ring. (B) A part of the Debye-Scherrer ring is recorded by the multiport charge-coupled device (MPCCD) detector for the bcc (002) plane of Ta at different times during the interaction. All the experimental images have the same color scale.



**Fig. 2. Experimental profiles of the stretching and postspallation compression in the Ta sample.** (A) Observation of the stretching in the experiment of the (002) plane of Ta using an azimuthal integration of the diffraction signal obtained onto the MPCCD detector (blue arrow). (B) Observation of the compression wave (purple arrow) due to the relaxation of the tension after spallation in the experiment of the (002) plane of Ta using an azimuthal integration of the diffraction signal obtained onto the MPCCD detector. The onset at the top left corresponds to the maximum stretching of the sample reached at a time  $t = 1725$  ps after the beginning of the interaction, whereas the onset at the top right corresponds to the dynamic fracture of the sample responsible for the generation of a compression wave propagating in the spall layer. The laser comes from the left, and the XFEL probe comes from the right. These illustrations are not to scale.

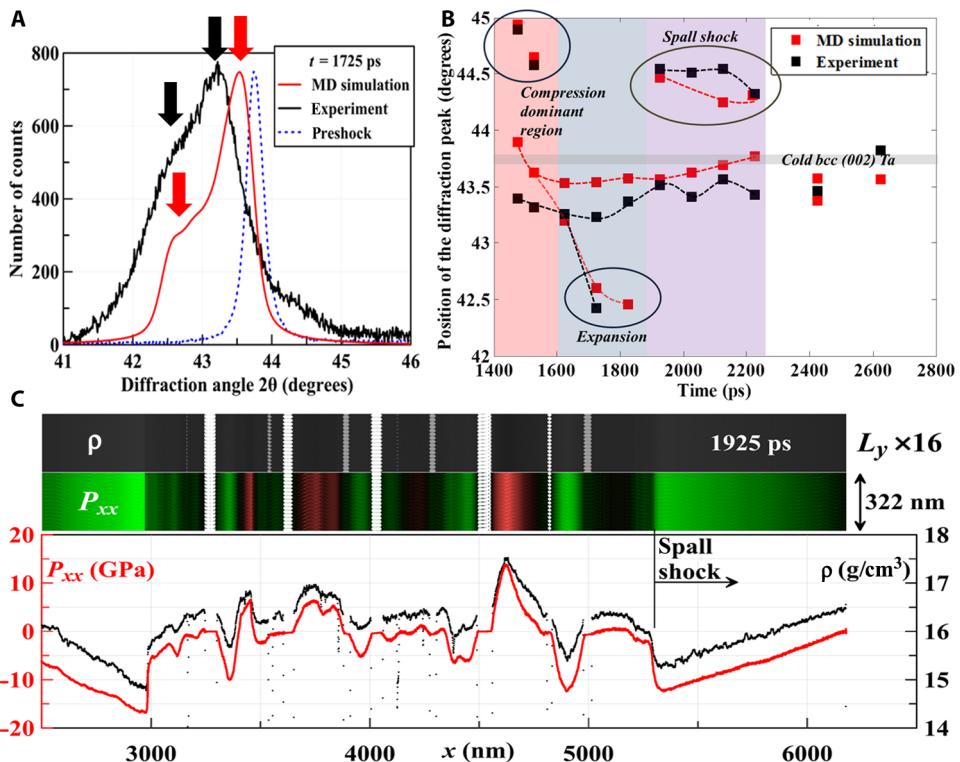
reached the free surface. These two phenomena of compression and expansion are contained in a very small space (that is, the region of the penetration depth of the x-rays) and time, which also demonstrates that a very sharp discrete rarefaction wavefront is created, propagating at  $\sim 4.5$  km/s.

At later times (for example,  $t = 1625$  ps), no lattices are compressed and one can only observe a peak from stretched lattices. The maximum expansion, which is related to the ultrafast fracture of the sample, occurs at  $t = 1725$  ps, where a second peak appears at  $2\theta \sim 42.42^\circ$ . The two positions of the maximum of the major peaks retrieved at that time have a spacing  $d = 1.679$  and  $1.709$  Å, which correspond to densities of  $\rho = 15.87$  and  $15.04$  g/cm $^3$ , respectively. Those densities correspond to the stretched solid states with respective pressures of  $-8.7$  and  $-16.78$  GPa according to the Birch-Murnaghan equation of state (see the Supplementary Materials) (28–30). The later times, that is,  $t \geq 1925$  ps, unambiguously show the progressive emergence of a compression wave (see Fig. 2B) with the appearance of a peak at  $2\theta \sim 44.60^\circ$ . This compression wave, referred to as the “spall shock” in the following, is generated by ultrafast void nucleations and propagates from the void locations toward the free surface of the spalled layer. The sudden stress relaxation from the negative to ambient pressure in the stretched lattice zone gives rise to the emergence of a strong recompression wave, which propagates in the spalled layer of the material. As a consequence, this spall shock is a strong signature of an initiation of the ultrafast fracture of the sample. The maximum compression due to the spall shock is observed at  $t = 2125$  ps, where the amplitude of the peak at  $2\theta \sim 44.60^\circ$  is maximized. Finally, at the latest probing time ( $t = 2625$  ps), the peak is almost at the initial position, although it is wider, probably due to a distribution of different strain (positive or negative) and residual temperature effects.

The experimental data allow us to directly determine the strain rate  $\dot{\epsilon} = \partial\epsilon/\partial t$ , where  $\epsilon = (d(t) - d_0)/d_0$ , with  $d(t)$  corresponding, in our case, to the spacing  $d$ , assuming a uniaxial deformation of the sample, and  $d_0$  to the initial  $d$  spacing. Before spallation (that is,  $t = 1725$  ps), the averaged elongation of the material is  $d(t = 1725$  ps) = 1.7094 Å and  $d_0(t = 1625$  ps) = 1.679 Å. The temporal dynamics shown in Fig. 2A inform us that the elongation from  $d_0$  to  $d(t)$  takes place within 100 ps. Therefore, it is only possible to give a lower limit of the experimental strain rate using this method, which should be  $\dot{\epsilon} > 1.81 \times 10^8$  s $^{-1}$ .

A large-scale MD simulation of a single-crystal Ta film was performed to directly compare and interpret the experimental results (see the Supplementary Materials for more details). A movie of the MD simulation is presented in the Supplementary Materials. At early time points, the MD simulation shows the propagation of the rarefaction wave from the free rear-side surface back into the sample, implying the emergence of tension and a lower density than the initial one. A direct comparison of diffracted x-ray signals coming from the MD simulation and experimental data is displayed in Fig. 3 (A and B). The overall trend (compare Fig. 3B) is well reproduced by the MD simulation, where the time evolution of the diffraction angle during expansion of the lattice and the propagation of the spall shock are in agreement. Therefore, the physical mechanism of the dynamic fracture of the sample is given by the MD simulation.

The dynamic fracture of the sample starts at  $t = 1800$  ps. The spall strength then corresponds to the tension (negative pressure) present just before the spallation occurs, that is,  $\sim -18$  GPa (see the Supplementary Materials). This stress in the rarefaction wave is reached after  $t = 1800$  ps in a wide fracture zone with length of about 1700 nm, where many nanovoids start to nucleate independently, as can be seen in Fig. 3C.



**Fig. 3. MD simulation and direct comparison with experimental data.** (A) Comparison of the diffraction signal obtained from the experiment and simulated by the MD just before spallation, where the stretching of the lattice is the most important at 1725 ps after the beginning of the interaction. The black and red arrows indicate the position of the maximum of the diffraction peaks. (B) Direct comparison between the position of the maximum of the different diffraction peaks in the experiment and in the simulation ( $t = 0$  being defined in the same manner in both cases). (C) Two-dimensional (2D) maps shown in the middle panel corresponding to the spatial distribution of density  $\rho(x,y)$  and the longitudinal component of the pressure tensor ( $P_{xx} \equiv -\sigma_{xx}$ ) are built by cloning the simulated narrow sample with  $L_y = 20$  nm by a factor of 16. White gaps correspond to pores or voids. For the  $P_{xx}$  map, the green color represents negative pressure and the red color represents positive pressure. The corresponding profiles of density and pressure  $P_{xx}$  at a time of 1925 ps after the beginning of the interaction are displayed in the bottom panel.

Because the voids appear with a time spread of  $\sim 10$  ps and each new void relaxes the surrounding tensile stress, only one void per about 150 nm can survive and grow further. Because of the periodical boundary condition imposed along  $y$  and  $z$  axes, the growth of voids is limited by the size of the MD simulation ( $L_{y,z} = 20$  nm). Some of them lead to multiple fractures of the sample, whereas others, which stop increasing in size while still smaller than 20 nm, are just closed pores (illustrated in gray color in Fig. 3C). The ultrafast fracture of the sample generates a shock wave (spall shock) starting from the location of the voids, which propagates through the spalled layer (which starts at  $x \sim 5000$  nm in Fig. 3C). The spall shock pressure and density profile exhibit almost a “plateau” of  $\sim 200$  nm with an amplitude of  $\sim 10$  GPa ( $\sim 17.2$  g/cm $^3$ ; see fig. S3B). Some material at the front of the shock wave still remains at a negative pressure ( $\leq -1$  GPa) because of tensile stresses. The spall shock compresses the sample up to a maximum density of  $\sim 17.5$  g/cm $^3$ , which corresponds to a pressure of  $\sim 15$  GPa (see the Supplementary Materials).

The strain rate is determined from the mass velocity profile  $u(x)$  obtained from the MD simulation (see the Supplementary Materials) and gives  $\dot{\epsilon} = 3.5 \times 10^8$  s $^{-1}$  just before the first rightmost spallation, that is, at a time of  $t = 1788$  ps after the beginning of the interaction. It is consistent with the lower limit calculated using the experimental data  $\dot{\epsilon} > 1.81 \times 10^8$  s $^{-1}$ , and it is also possible to obtain a “pseudo-experimental strain rate” using (i) the elongation of the sample given by the experimental data and (ii) the time  $t$  of the fracture observed in the MD

simulation. In that case, the pseudo-experimental strain rate is  $\dot{\epsilon} \sim 2.9 \times 10^8$  s $^{-1}$ , again in excellent agreement with the MD simulation.

In conclusion, we have demonstrated the feasibility of recording the evolution of material at extreme deformation rates  $\dot{\epsilon} \sim 2 \times 10^8$  to  $3.5 \times 10^8$  s $^{-1}$  using a real-time x-ray monitoring technique, opening the way to investigating the dynamic fracture of materials at the lattice level and revealing the atomic structure under tension. Using this technique, a maximal density decrease of 8 to 10%, associated with the onset of spallation in a tantalum sample, was directly measured. Thus, a spall strength of  $\sim 16.8$  GPa was calculated. This direct method should provide a more accurate spall strength than one estimated from the measured pullback velocity of the rear-side boundary of the spalled layer (31), allow the study of dynamical fracture (spallation) of a material at ultrahigh strain rate and atomic scale, and provide a way to accurately benchmark MD simulation. This is a crucial point for future technological and advanced material creation.

The experimental data show extremely good agreement with both previous results and our MD simulation (see the Supplementary Materials), which not only paves the way toward the direct measurement of spall strength of materials as a function of strain rate but also highlights the usefulness of these facilities to investigation of high-speed crack dynamics and uncommon stress-induced solid-solid phase transitions (32). These transitions could have a significant impact in the industry to develop new materials, allowing interesting mechanical

properties for the development of spacecraft, satellite, and plasma-facing components in nuclear power plant facilities.

## MATERIALS AND METHODS

The experiment was carried out on the beamline 3 (BL3) at SACLÀ. The spectrum of the x-ray beam was recorded on-shot to minimize uncertainties related to the evaluation of the crystallographic lattice spacing  $d$  of the sample. The optical laser beam was focused on the target using a  $f = 500$  lens. The next step was to defocus it to have a focal spot larger than the one of the x-ray beam. The synchronization of the main optical beam with the XFEL beam was performed using the following procedure: Initially, we measured the time difference between the optical laser and XFEL beams with a photodiode from a polyimide sample at the target center chamber. To have a precision of  $\sim 25$  ps for the beginning of pulse, we used a specially designed target made of a 500-nm-thick polycrystalline Au foil coating deposited on a 100- $\mu\text{m}$ -thick acrylic substrate. The optical beam irradiated the Au side of the target, and the XFEL beam probed the spacing  $d$  (in measuring the  $2\theta$  angle) through the acrylic [for more details, see Hartley *et al.* (33)]. The delay between the optical and the XFEL was then changed to observe when the Au lines became broadened for the first time due to sample heating. In that case, we could evaluate a typical laser intensity that corresponded to our time  $t = 0$  (see the Supplementary Materials).

## SUPPLEMENTARY MATERIALS

Supplementary material for this article is available at <http://advances.sciencemag.org/cgi/content/full/3/6/e1602705/DC1>

fig. S1. Single-shot x-ray spectra of Ta plasma irradiated by the high-power optical laser.

fig. S2. Pulse shape of the optical beam at target center chamber.

fig. S3. Velocity of the Lagrangian particle provided by hydrodynamic (MULTI) modeling.

fig. S4. Comparison between the new EAM Ta potential and experimental shock Hugoniot curve.

fig. S5. Shadowgraph of mass distribution after nucleation of first voids in the MD-simulated Ta sample.

fig. S6. MD simulation of the experiment.

fig. S7. Determination of the strain rate from the flow velocity profile just before nucleation of the first voids in the MD simulation.

fig. S8. Determination of the strain rate from the flow velocity profile just before nucleation of the first voids in the MD simulation.

fig. S9. Direct comparison between experimental profiles and x-ray profiles derived from the MD simulation.

fig. S10. Experimental determination of the position of the different peaks at  $t = 1725$  ps using the Gaussian method.

fig. S11. Experimental determination of the position of the different peaks at  $t = 2125$  ps using the Gaussian method.

fig. S12. Experimental determination of the position of the different peaks at  $t = 2125$  ps using the Lorentzian method.

fig. S13. Spall strength versus strain rate for tantalum.

table S1. Pressure and density retrieved from the experimental results at  $t = 1725$  ps displayed in fig. S10.

table S2. Pressure and density retrieved from the experimental results at  $t = 2125$  ps displayed in fig. S11.

movie S1. Evolution of density and pressure in the sample given by MD simulation.

movie S2. Experimental data obtained at SACLÀ.

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