Supplementary information

A statistical analysis of the second 'pop-in' behaviour of the spherical-tip nanoindentation of Zr-based bulk metallic glasses

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a	Contact radius
P	Indenter load
h	Indentation depth
R_i	Spherical tip radius
E_r	Reduced modulus
E_s, E_i	Elastic modulus of sample and indenter
ν_s, ν_i	Poisson ratio of sample and indenter
p_m	Mean contact pressure
p_o	Mean pressure of contact
τ_{max}	Maximum shear stress at first pop-in
τ_y	Local shear yield strength
τ^{SP}	Maximum shear stress at second pop-in load
τ_{max}	Maximum shear stress at first pop-in
$\Delta\tau_{max}$	Stress drop associated with initiation of first shear band
τ_2	Stress created solely due to the second shear band
s	Vector representing a collection of sample sizes

s_l	l^{th} sample size in s
m	Maximum sample size
N	Number of random samples selected for each sample size
U	Dataset
u	Data point
u_i	i^{th} data point in u
θ_0	Initial parameter space
$f(u)$	Probability density function
$\theta_N^{\widehat{[s_l]}}$	Best fitting parameters for N random samples of each sample size
$\theta_{mean}^{[s_l]}$	Mean of parameter space for each sample size
$\theta_{var}^{[s_l]}$	Variance of parameter space for each sample size
U	Dataset
θ	Parameter space for $f(x)$
μ	Mean of a Gaussian distribution
σ	Standard deviation of a Gaussian distribution
$L(.)$	Likelihood function
$\hat{\theta}$	Best fitting parameters
n	Total number of data points or number of indents on the sample
γ	Total number of model parameters
θ_γ	γ^{th} parameter of a distribution
$\widehat{(.)}$	Maximum Likelihood Estimate of any parameter, such as θ, μ, σ
α	Location parameter of Weibull distribution
β	Scale parameter of Weibull distribution
m	Weibull modulus
$f_M(u)$	Density function of a mixture model
θ_{Md}	d^{th} components in a mixture model
p	Proportion of a component distribution in a mixture model
t_{id}	Probability that observation i belongs to component d of the mixture model
H_o	Null hypothesis
α_s	Significance level

D_{crit}	Critical value of D
p-value	Threshold value of α_s
r, z	Spatial co-ordinates for stress contours
v	Displacement
σ_r	Radial stress
σ_θ	Hoop stress
σ_z	Normal pressure directly beneath the indenter
τ_{rz}	Shear stress
$\sigma'_r, \sigma'_\theta, \sigma'_z, \tau'_{rz}$	Normalized stresses
$\sigma_{1,3}$	Principal stresses in r-z plane
σ'_m	Hydrostatic stress
P_{FP}	First pop-in load
P_{SP}	Second pop-in load
$\dot{P}$	Loading rate

S1. Shear band trajectories and Hertzian contact relations

The classical Hertz contact theory relates the contact radius, a, to the indenter load, P, the indenter radius, R_i, and the elastic properties of the material using the following equation [1]:

$$a = \left(0.75\frac{PR_i}{E_r}\right)^{1/3} \quad \text{(S1)}$$

where E_r is reduced modulus which accounts for deformation in both specimen and indenter, and can be given as:

$$\frac{1}{E_r} = \frac{1-\nu_s^2}{E_s} + \frac{1-\nu_i^2}{E_i} \quad \text{(S2)}$$

where E_s and E_i are elastic modulus of sample and indenter. ν_s and ν_i are Poisson ratio of sample and indenter respectively.

In spherical indentation, the maximum shear stress at the first pop-in, τ_{max}, represents the critical shear stress required for the onset of plasticity in the indented material. τ_{max} occurs directly below the rotational axis at a distance approximately half the contact radius and is given as [2,3]:

$$\tau_{max} = 0.31p_o = 0.47p_m = 0.47 * \left(\frac{4}{3\pi}\right)\frac{E_r}{R_i^{1/2}}h^{1/2} \tag{S 3}$$

where h and p_o are indentation depth and maximum pressure of contact. At P_{FP}, which is the first plastic event, τ_{max} corresponds to the local shear yield strength, τ_y. Similarly, τ_{max} at P_{SP} is the shear stress, τ^{SP} that represents the second plastic event. Unlike τ_y, which represents the stress that triggers incipient plasticity, τ^{SP} is not the actual stress at which the second plastic event is initiated. This is because there is some stress relaxation after the first pop-in, which causes a drop in the mean contact pressure, p_m.

The p_m can be calculated by dividing both sides of eq. (S1) by contact area, $A = \pi a^2 = \pi R_i h$, and can be written as:

$$p_m = \frac{P}{\pi a^2} = \frac{4}{3\pi}\frac{E_r}{R_i^{1/2}}h^{1/2} \tag{S4}$$

The drop in mean contact pressure is written below:

$$\Delta p_m = \frac{4}{3\pi}\frac{E_r}{{R_i}^{1/2}}(\Delta h)^{1/2} \tag{S5}$$

The stress drop, $\Delta\tau_{max}$, associated with p_m is:

$$\Delta\tau_{max} = 0.47 * \left(\frac{4}{3\pi}\right)\frac{E_r}{R_i^{1/2}}\Delta h^{1/2} \tag{S6}$$

where Δh is the magnitude of the first pop-in displacement.

Finally, the actual maximum shear stress for initiating the second plastic event, τ_2, can be estimated by subtracting $\Delta\tau_{max}$ from the stress at which second pop-in event, τ^{SP}, occurs and can be written as:

$$\tau_2 = \tau^{SP} - \Delta\tau_{max} \tag{S7}$$

The stress field underneath the spherical indenter are:

$$\sigma'_r = \frac{\sigma_r}{p_m} = \frac{3}{2}\left\{\frac{(1-2\nu)a^2}{3r^2}\left[1-\left(\frac{z}{\sqrt{v}}\right)^3\right] + \left(\frac{z}{\sqrt{v}}\right)^3 \frac{a^2 v}{v^2 + a^2 z^2} + \frac{z}{\sqrt{v}}\left[v\frac{(1-\nu)}{a^2+v} + \frac{(1+\nu)\sqrt{v}}{a} tan^{-1}\left(\frac{1}{\sqrt{v}}\right) - 2\right]\right\} \tag{S 8}$$

$$\sigma'_\theta = \frac{\sigma_\theta}{p_m} = -\frac{3}{2}\left\{\frac{(1-2\nu)a^2}{3r^2}\left[1-\left(\frac{z}{\sqrt{\mathrm{v}}}\right)^3\right] + \frac{z}{\sqrt{\mathrm{v}}}\left[2\nu + v\frac{(1-\nu)}{a^2+\mathrm{v}} - \frac{(1+\nu)\sqrt{\mathrm{v}}}{a}\tan^{-1}\left(\frac{a}{\sqrt{\mathrm{v}}}\right)\right]\right\} \tag{S 9}$$

$$\sigma'_z = \frac{\sigma_z}{p_m} = -\frac{3}{2}\left(\frac{z}{\sqrt{\mathrm{v}}}\right)^3\left(\frac{a^2\mathrm{v}}{\mathrm{v}^2 + a^2 z^2}\right) \tag{S 10}$$

$$\tau'_{rz} = \frac{\tau_{rz}}{p_m} = -\frac{3}{2}\left(\frac{rz^2}{\mathrm{v}^2 + a^2 z^2}\right)\left(\frac{a^2\sqrt{\mathrm{v}}}{a^2 + \mathrm{v}}\right) \tag{S 11}$$

where v is the displacement, defined as

$$\mathrm{v} = 0.5\left[r^2 + z^2 - a^2 + \sqrt{(r^2+z^2-a^2)^2 + 4a^2z^2}\right] \tag{S 12}$$

The principal stresses in the rz plane are given by:

$$\sigma_{1,3} = \frac{\sigma_r + \sigma_z}{2} \pm \sqrt{\left(\frac{\sigma_r - \sigma_z}{2}\right)^2 + \tau_{rz}^2} \tag{S 13}$$

$$\sigma_2 = \sigma_\theta \tag{S 14}$$

Maximum shear stress,

$$\tau_{max} = \frac{1}{2}(\sigma_1 - \sigma_3) \tag{S 15}$$

Therefore,

$$\tau_{max} = \sqrt{\left(\frac{\sigma'_r - \sigma'_z}{2}\right)^2 + {\tau'_{rz}}^2} \tag{S 16}$$

Hydrostatic stress,

$$\sigma'_m = \frac{\sigma'_r + \sigma'_\theta + \sigma'_z}{3} \tag{S 17}$$

Using eqs. S29- S38, four different stress trajectories of τ_{max} are plotted in Fig. 3. These contours, A, B, C and D represent 2-D projection of potential shear planes where plastic deformation may occur in metallic glasses (MGs). The contours are drawn based on the Mohr-Coulomb yield criterion, which has been found to capture the pressure sensitivity of plastic flow in MGs [1,9,13–19].

S 2. Statistical procedures employed

S2.1 Sample size optimization procedure:

In this procedure, the aim is to choose a sample size that is large enough to provide accurate and precise estimates of the population parameters of interest. The procedure followed is mentioned below:

1. A vector, $s = [s_1, s_2, s_3, \ldots, s_m]$, representing a collection of sample sizes, s_l, is assumed.
2. For each s_l, N random samples are generated from the probability distribution, $f(u|\theta_0)$, where u and θ_0 represent data and initial parameter space, respectively.
3. Then, the best fitting parameters for all N random samples of s_l, $\widehat{\theta_N^{[s_l]}}$, are determined.
4. Finally, the mean, $\theta_{mean}^{[s_l]}$, and variance, $\theta_{var}^{[s_l]}$, of parameter space, for each s_l, are calculated using eqs. S7 and S8:

$$\theta_{mean}^{[s_l]} = \frac{1}{N}\sum_{J=1}^{N} \widehat{\theta_J^{[s_l]}} \tag{S 18}$$

$$\theta_{var}^{[s_l]} = \frac{1}{N}\sum_{J=1}^{N} (\widehat{\theta_J^{[s_l]}} - \theta_0)^2 \tag{S 19}$$

As the sample size increases, $\theta_{mean}^{[s_l]}$ tend to θ_0 and $\theta_{var}^{[s_l]}$ approaches zero asymptotically. The s_l having relatively small $\theta_{var}^{[s_l]}$ correspond to the optimum sample size for the chosen statistical model.

The sample size optimization test is conducted for all the considered uni- and bi-modal statistical distributions which include Gausian, Lognormal, 2-parameter and 3-parameter Weibull. An illustrative example of sample size optimization test for a 3-parameter unimodal Weibull distribution is depicted in Fig. S1. On conducting optimization tests for all the statistical models considered in this study (both single component and mixture), a sample size of 50 is found reasonably significant for all the models considered [4–6].

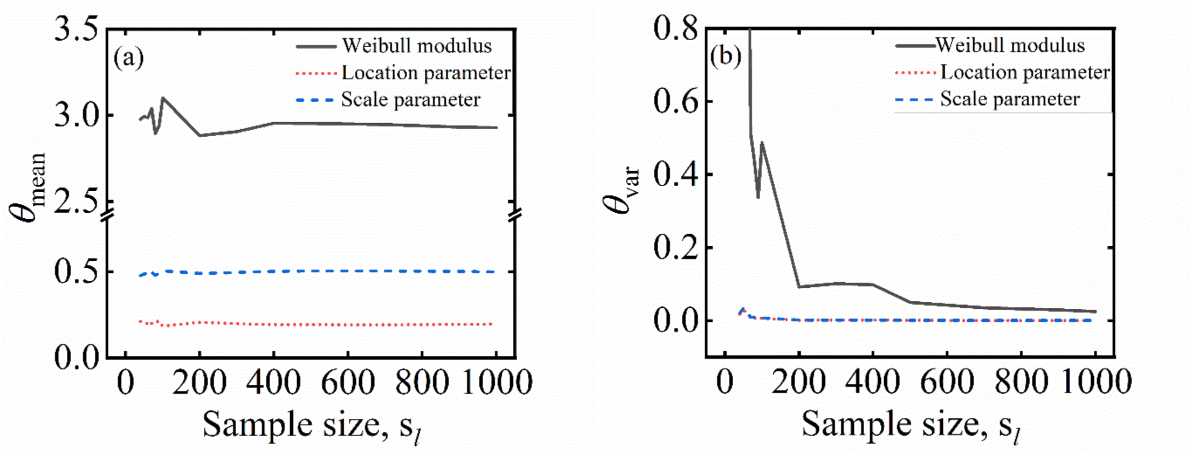


Fig. S1. Variations of (a) θ_{mean} and (b) θ_{var} with the sample size, s_l for the different parameters of 3-parameter Weibull model (θ_0: Location parameter, α = 0.2, Scale Parameter, β = 0.5, Weibull Modulus, m = 3). θ_{var} is negligible for all values of s_i > 50 for all the sample optimization tests conducted in this study.

S2.2. Statistical inference through maximum likelihood (ML) approach:

Maximum Likelihood Estimation (MLE) is a statistical tool, used to estimate the best fitting parameters of a probability distribution by maximizing the likelihood of obtaining the observed data. For this, a dataset, $U = \{u\}$ is assumed where each data point is represented by u_i. U is fitted with an arbitrary probability density function (PDF), $f(u|\theta)$, where θ represents the parameter space. For illustration, the Gaussian distribution, for fitting the dataset, is chosen, which describes the probability density function as:

$$f(u) = \left(\frac{1}{\sigma\sqrt{2\pi}}\right) exp\left(-\frac{(u-\mu)^2}{2\sigma^2}\right) \tag{S 20}$$

where μ and σ denote mean and standard deviation, respectively [7,8]. Note that, μ and σ represent parameter space, θ, for Gaussian distribution. To find the best fitting parameters or MLEs ($\hat{\theta}$), the ML approach needs to be followed. The likelihood function, $L(\theta)$, can be defined as follows,

$$L(\theta) = \Pi_{i=1}^{n} f(u_i|\theta) \tag{S 21}$$

where n is the total number of data points or the number of indents on the sample. The global maxima of $L(\theta)$ yield the best fitting parameters, $\hat{\theta}$.

S2.2.1. Concept of Profile Likelihood:

The data $\{u_1, u_2, u_3, \ldots, u_n\}$ is assumed to follow a probability distribution with unknown parameters $\theta = (\theta_1, \theta_2, \theta_3, \ldots, \theta_\gamma)$. The likelihood function, $L(\theta|u)$, is the product of the PDF evaluated at each observed data point and can be written as:

$$L(\theta|u) = f(u_1, u_2, u_3, \ldots.., u_n|\theta) \tag{S 22}$$

The profile likelihood for a parameter of interest, say θ_1, is defined as the maximum value of the likelihood function when θ_1 is fixed at a particular value and remaining parameters $(\theta_2, \theta_3, \ldots, \theta_\gamma)$ are allowed to vary. Mathematically, it can be expressed as:

$$\hat{\theta} = argmax_{\theta_1} L(\theta_2|u) \tag{S 23}$$

where argmax denote the value of θ_2 that maximizes the likelihood function [7–9].

S2.2.2. MLE for single component distribution functions

<u>MLE for Gaussian distribution:</u>

The probability density functions (PDFs) for the Gaussian distribution, $f(u)$, is given by:

$$f(u) = \left(\frac{1}{\sigma\sqrt{2\pi}}\right) exp\left(-\frac{(u-\mu)^2}{2\sigma^2}\right) \tag{S 24}$$

where u is the observed data, μ is the mean and σ is the standard deviation of the distribution

The MLE for the mean and variance of a Gaussian distribution can be obtained by maximizing the likelihood function, $L(\mu, \sigma|u)$ with respect to the parameters μ and σ.

The log-likelihood function can be written as:

$$lnL(\mu, \sigma|u) = -\frac{n}{2}ln(2\pi) - \frac{n}{2}ln(\sigma^2) - \frac{\sum(u_i - \mu)^2}{2\sigma^2} \quad \text{(S 25)}$$

To find the MLE, the log-likelihood function is differentiated with respect to μ and σ. The MLE for mean, $\hat{\mu}$, :

$$\hat{\mu} = \frac{1}{n}\sum_{i=1}^{n} u_i \quad \text{(S 26)}$$

and the MLE for the standard deviation, $\hat{\sigma}$, is:

$$\hat{\sigma} = \sqrt{\left(\frac{1}{n}\right)\sum_{i=1}^{n}(u_i - \hat{\mu})^2} \quad \text{(S 27)}$$

MLE for Lognormal distribution:

The PDF for the lognormal distribution, $f(u)$, is given by:

$$f(u) = \left(\frac{1}{u\sigma\sqrt{2\pi}}\right) exp\,(-\frac{(ln(u) - \mu)^2}{2\sigma^2}) \quad \text{(S 28)}$$

The MLE for mean, $\hat{\mu}$, and standard deviation, $\hat{\sigma}$, are:

$$\hat{\mu} = \frac{1}{n}\sum_{i=1}^{n} ln(u_i) \quad \text{(S 29)}$$

$$\hat{\sigma} = \sqrt{\left(\frac{1}{n}\right)\sum_{i=1}^{n}(\ln(u_i) - \hat{\mu})^2} \quad \text{(S 30)}$$

MLE for 2-parameter Weibull distribution:

The PDF for the 2-parameter Weibull distribution, $f(u)$, is given by:

$$f(u) = \begin{cases} \frac{m}{\beta}\left(\frac{u}{\beta}\right)^{m-1} \exp\left(-\left(\frac{u}{\beta}\right)^{m}\right), & u \geq 0 \\ 0, & u < 0 \end{cases} \quad \text{(S 31)}$$

where m and β are Weibull modulus and scale parameter of the distribution. Taking the logarithm of the likelihood function and setting its derivative with respect to β and m to zero leads to a system of non-linear equations, as mentioned below:

$$\frac{1}{\widehat{m}} - \frac{\sum_{i=1}^{n} u_i{}^{\widehat{m}} \ln u_i}{\sum_{i=1}^{n} u_i{}^{\widehat{m}}} + \frac{\sum_{i=1}^{n} \ln u_i}{n} = 0 \quad \text{(S 32)}$$

$$\hat{\beta}^{\widehat{m}} = \frac{\sum_{i=1}^{n} u_i{}^{\widehat{m}}}{n} \quad \text{(S 33)}$$

These non-linear equations do not have a closed form solution, and thus cannot be solved analytically. Numerical methods such as the Regula Falsi method is typically used to obtain the MLEs of m.

MLE for 3-parameter Weibull distribution:

The PDF for the 3-parameter Weibull distribution, $f(u)$, is given by:

$$f(u) = \begin{cases} \frac{m}{\beta}\left(\frac{u-\alpha}{\beta}\right)^{m-1} exp\left(-\left(\frac{u-\alpha}{\beta}\right)^{m}\right), & u \geq 0 \\ 0, & u < 0 \end{cases} \quad \text{(S 34)}$$

where α, β, m are location parameter, scale parameters and Weibull modulus respectively. $\hat{\beta}$ and $\widehat{m}$ are estimated using eq. S21 and S22. $\hat{\alpha}$ can be estimated by computing the profile likelihood of α in [0, u_1] discretized in steps of 0.01.

S2.2.3. MLE for mixture models via Expectation–Maximization (EM) Algorithm

A mixture model is a statistical model that represents the presence of different sub-populations within an overall population. The PDF for the two component mixture models, f_M, is obtained by weighted linear combination of each component, as following:

$$f_M(u) = pf(u|\theta_{M1}) + (1-p)f(u|\theta_{M2}) \tag{S 35}$$

where p is the mixing proportions: $0 < p < 1$, and θ_{M1}, θ_{M2} are the model parameter for a mixture model.

Since $f_M(u)$ is a weighted combination of the two component distributions, it has twice as many parameters as the component models in addition to p. Therefore, the task of maximizing the likelihood function of $f_M(u)$ over such a parameter space is challenging. This problem of MLE is assuaged by EM algorithm, which provides a computationally efficient way to estimate parameters of complex models.

The EM algorithm is an iterative algorithm used to estimate the parameters of a probabilistic model when the observed data is incomplete or contain missing values. In the present study, U is deemed incomplete as it is unknown which mixture component u belongs to [9].

The EM algorithm for MLE of two component mixture models proceeds as follows:

1. Initialize the parameters:

   The parameters θ_{M1}, θ_{M2} are initialized by setting them to some arbitrary values.

2. Expectation step (E-step):

In this step, the posterior probability of each observation belonging to each component is computed. Let t_{id} be the probability that observation i belongs to component d of the mixture model. Then we compute t_{id} using Bayes' theorem as mentioned below:

$$t_{id} = \frac{p_d f_d(u_i|\theta_{Md})}{\sum_{d=1}^{2} p_d f_d(u_i|\theta_{Md})} \tag{S 36}$$

where p_d is the mixing proportion (i.e., weight) of the component d.

3. Maximization step (M-step):

   In this step, the estimated parameters are updated based on the posterior probabilities computed in the E-step.

$$p_d = \frac{1}{n}\sum_{i=1}^{n} t_{id} \tag{S 37}$$

4. Repeat steps 2 and 3 until convergence, where the convergence is typically determined by monitoring the change in the log-likelihood or the model parameters between iterations.

5. Once convergence is reached, the final estimates of the model parameters are obtained.

S2.3. Akaike Information Criterion (AIC)

The AIC is a statistical measure utilized to assess the suitability of the selected model to describe the data. The AIC is defined as follows:

$$\text{AIC} = -2lnL(\hat{\theta}) + 2\gamma \tag{S 38}$$

where $L(\hat{\theta})$ is the maximum likelihood and γ is the number of independent parameters in the model, which are listed in Table S1. *The model that yields the lowest AIC for a given dataset is the best fitting model for that particular dataset.* However, it should be noted that the AIC is only one of several criteria that can be used for model selection, and it should not be used in isolation to make decisions about the best model [10].

Table S1: Number of independent parameters in different models

Model	Number of independent parameters, γ	Name of independent parameters
Gaussian	2	Mean, Standard deviation
Lognormal	2	Mean, Standard deviation
2-parameter Weibull	2	Shape, Scale
3-parameter Weibull	3	Shape, Scale, Threshold

S2.4. Kolmogorov-Smirnov (KS) test

To select the best fit distribution from uni- and bi-modal 3W distributions, three goodness-of-fit tests (Kolmogorov-Smirnov (KS), Anderson-Darling (AD), Chi-squared (CS)) are commonly utilized at a significance level (α_s) of 0.05. Out of all three, we have employed the 'bootstrap method' of KS test as it is exact and the statistic is independent of the underlying cumulative distribution function.

In this approach, the cumulative distribution function of the distribution is compared to the empirical distribution function of the data. The empirical distribution function of the given data $\{u_1, u_2, \dots\dots, u_n\}$ can be described as follows:

$$f_n(u) = \frac{Number\ of\ u_i \leq u}{n} \tag{S 39}$$

The KS test is used to evaluate whether the data comes from null hypothesis (null hypothesis, H_o: the samples come from a particular distribution; alternative hypothesis: the samples do not come from a particular distribution) The p-value is the probability that the null hypothesis, is true. A smaller p-value (≤ 0.05) indicates that it is false while a higher p-value indicates that it is true. Note that the KS test is only appropriate for testing if the candidate models fit the experimental data well and should not be used for choosing the appropriate statistical model [11,12]. The AIC tests should be exclusively relied upon for model selection.

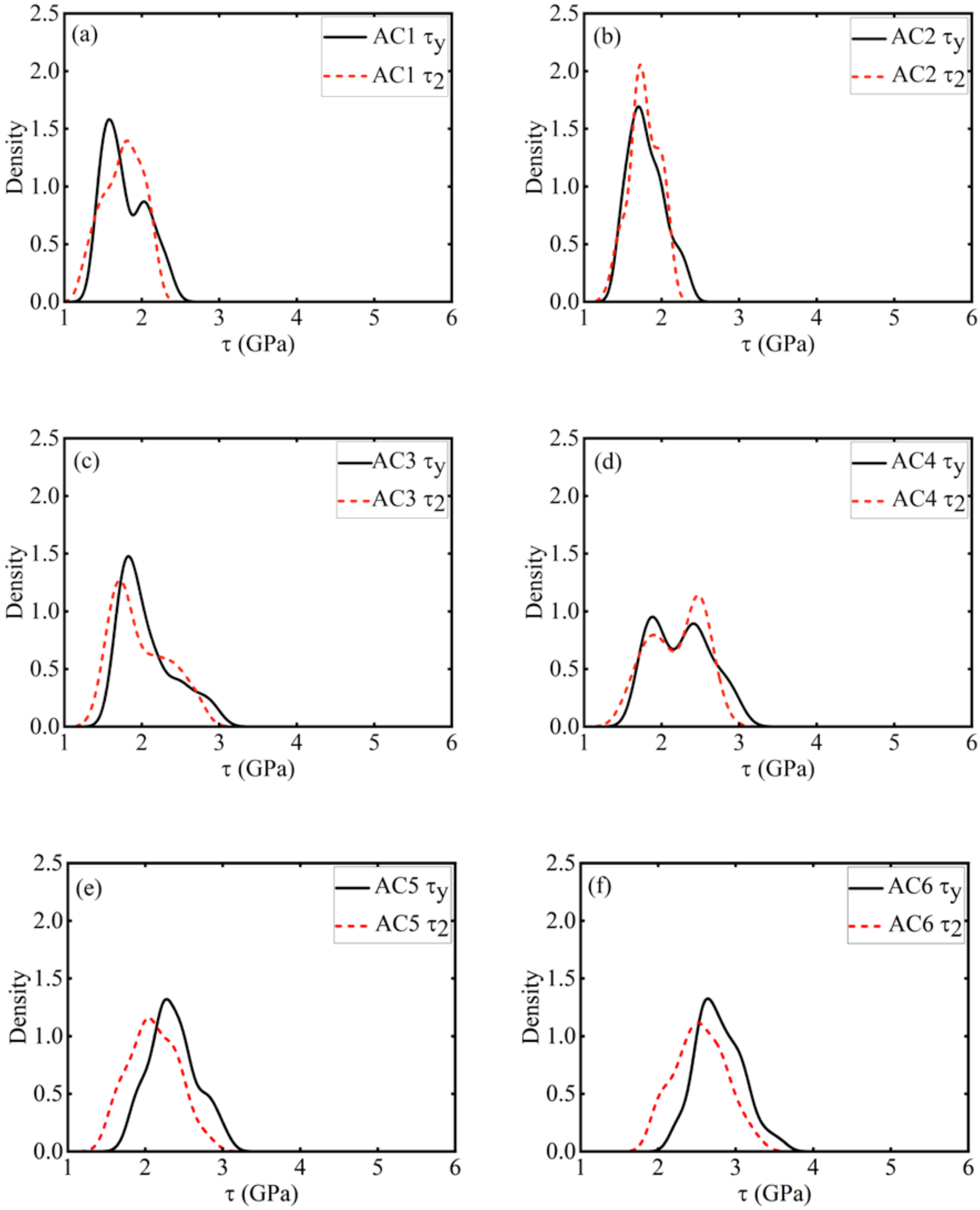
S3. Kernel density estimate plots

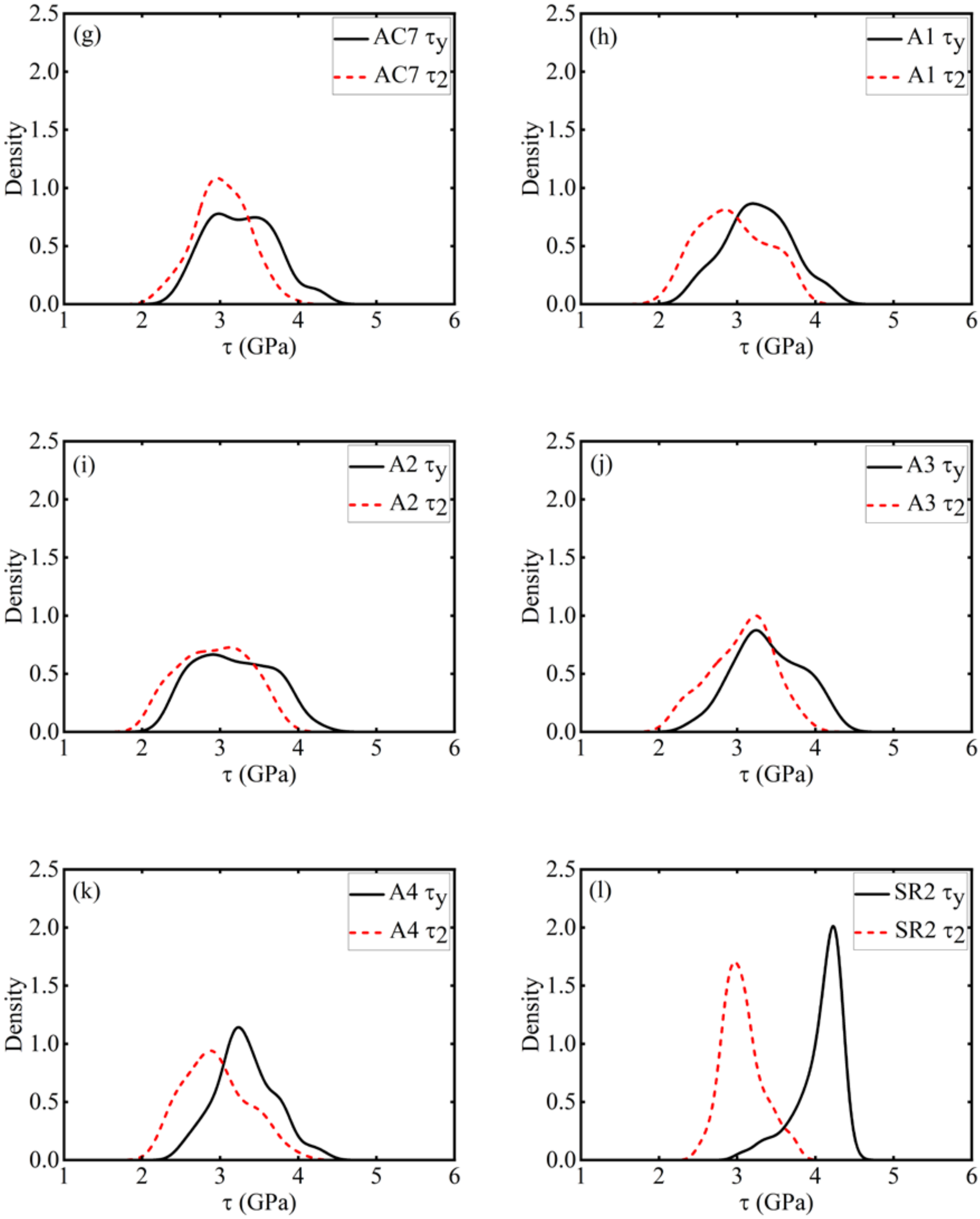
The shear stress ranges and KDE plots are provided in Table S1 and Fig. S2 respectively.

Table S2: Range of τ_y and τ_2

Sample	Range of τ_y (GPa)	Range of τ_2 (GPa)	% reduction in τ_y (lower bound)	% reduction in τ_y (higher bound)	Average % reduction in τ_y
AC1	1.39-2.41	1.25-2.17	10.07	9.96	10.01
AC2	1.44-2.33	1.32-2.14	8.33	8.15	8.24
AC3	1.66-2.99	1.47-2.75	11.44	8.02	9.74
AC4	1.67-3.04	1.5-2.82	10.17	7.24	8.71
AC5	1.72-3.07	1.55-2.82	9.88	8.14	9.01
AC6	2.21-3.59	2-3.3	9.50	8.08	9.50
AC7	2.46-4.29	2.20-3.9	10.57	9.09	9.83
A1	2.37-4.17	2.12-3.7	10.54	11.27	10.91
A2	2.39-4.23	2.17-3.8	9.20	5.94	7.57
A3	2.43-4.28	2.2-3.87	9.46	9.57	9.52
A4	2.51-4.33	2.25-3.97	10.35	7.24	8.81
SR2	3.02-4.48	2.52-3.75	16.5	16.29	16.39

SR3	3.4-5.3	2.85-4.48	16.17	15.47	15.82





(g)
AC7 τy
AC7 τ2
(h)
A1 τy
A1 τ2
(i)
A2 τy
A2 τ2
(j)
A3 τy
A3 τ2
(k)
A4 τy
A4 τ2
(l)
SR2 τy
SR2 τ2
Density
τ (GPa)

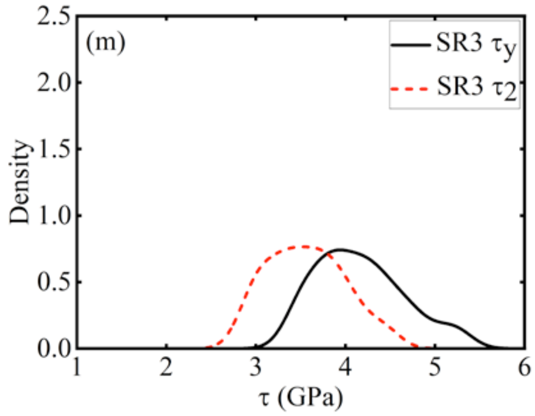


Fig. S2. Strength distribution represented as kernel density estimates (KDE) of 1st and 2nd pop-in.

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