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# Evaluation of Mesoscale Tension Tests for Rapid Screening of Mechanical Properties in High-Strength Steels

by Daniel Magagnosc, Michael Rupinen, and Daniel Field

# NOTICES

## Disclaimers

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**14. ABSTRACT**
Applications of miniaturized test methods are rapidly growing in the field of high-throughput materials discovery. When using small-scale test specimens, care must be taken to account for how specimen size affects the measured mechanical response. Here, the macroscale and mesoscale responses of a nominal HP 9-4-30 high-strength steel alloy are directly compared. The tensile behavior was qualitatively similar regardless of specimen size. However, measurable differences in strength and ductility were observed. These differences are attributed to extrinsic effects of the sample fabrication process and sample geometry. Recommendations to alleviate each of these effects are proposed.

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## 1. Introduction

Evaluating the mechanical properties of materials using small-scale test specimens has become a widely adopted practice. Numerous methods—including nanoindentation, micropillar compression, microscale tension, and cantilever bending—have been developed to extract material strength and fracture properties.[1–6] In metallic systems, micropillar and microscale tension testing are now routinely used for fundamental research on materials deformation mechanisms with minimal consumption of material. For instance, sample size effects on strength and plasticity have been widely investigated.[7–9] Additionally, microscale testing enables investigations of both intrinsically small-scale materials (e.g., materials micro-electromechanical systems)[10,11] and materials that are difficult to process into bulk test specimens.[12,13]

The shift towards high-throughput materials discovery[14,15] further emphasizes the utility of small-scale mechanical testing. Numerous frameworks for identifying new structural metals have been proposed.[16,17] A key element of these approaches is quickly screening mechanical properties. Often this is restricted to hardness-based techniques that do not provide information on ductility or fracture. Small-scale tension or compression tests may solve several challenges for materials discovery by combining high-throughput testing methods with higher-fidelity measurements.[18,19]

As the application of small-scale testing methods expands in the field of high-throughput materials discovery, practitioners must be cognizant of how miniaturization impacts property measurements, especially when extrapolations to bulk properties are desired. Several specimen size effects are well known. For instance, in flaw-dominated materials such as ceramics, reducing specimen size can increase the tensile strength.[12,20] Similarly, performing measurements at a length scale smaller than a critical microstructural feature may introduce significant stochasticity.[21]

Here, a direct comparison of tensile behavior in a high-strength steel is made between mesoscale and macroscale tests. While the tensile behavior is found to be comparable across the measured lengths scales, several key differences are observed. These property differences are assessed with respect to extrinsic factors that arise when miniaturizing the tensile specimens. Based on these results, recommendations for future test methods are identified.

## 2. Methods

A nominal HP 9-4-30 was tested in the final water-quenched and tempered condition, consisting of a tempered martensite microstructure. Macroscale tensile specimens were waterjet cut parallel to the plate rolling direction. The sample geometry followed the ASTM E8 subsize flat specimen geometry.[22] After cutting, tensile bars were surface ground to remove the decarburization layer. Mesoscale tension specimens were prepared by first cutting and grinding an approximately 1-mm-thick strip parallel to the plate rolling direction. Tensile specimens were then fabricated via conventional machining using a UMC-500SS Haas machining system and a 0.0625-inch four-fluted carbide end mill into the geometry shown in Fig. 1. After milling, the specimens were ground to a final thickness of less than 500 µm to ensure that the required forces were within the load capacity of the test frame.

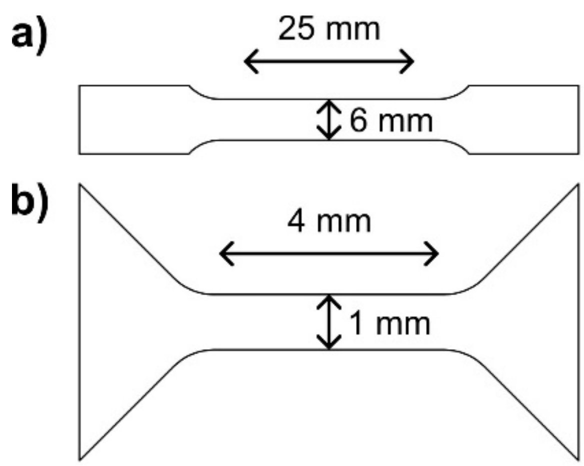


**Fig. 1 a) Macroscale and b) mesoscale sample geometries**

Macroscale tension tests were performed using an Instron 5985 electromechanical universal testing system. Tests were conducted at a nominal strain rate of $7.8 \times 10^{-4}$ $s^{-1}$ with an applied displacement rate of 0.5 mm/min. Strains were determined using 3D digital image correlation (DIC). For DIC, the specimens were painted with a white base coat and speckled with black spray paint. Strains were then calculated using the Vic3D software.

Mesoscale tension tests were performed using a Psylotech µTS universal load frame equipped with a 1.6-kN load cell and integrated stereo microscope. Tests were performed at a nominal strain rate of $1.25 \times 10^{-3}$ $s^{-1}$ with an applied displacement rate of 300 µm/min. Stereo image pairs were captured using the stereomicroscope for determining strains using DIC. The samples were painted with a white base coat and speckled using black toner powder. Strains were calculated using Vic3D.

## 3. Results

Figure 2 presents the stress–strain response and evolution of the strain hardening rate for both the mesoscale and macroscale tensile specimens. Regardless of the specimen size, the stress–strain response follows a similar shape. Following yield, the strain hardening rate continuously decreases with increase strain and the strain hardening behavior is remarkably consistent regardless of specimen size. In contrast, the strength and elongation behavior show distinct differences. The yield and tensile strengths are higher for the mesoscale while the total elongation is decreased versus the macroscale specimens.

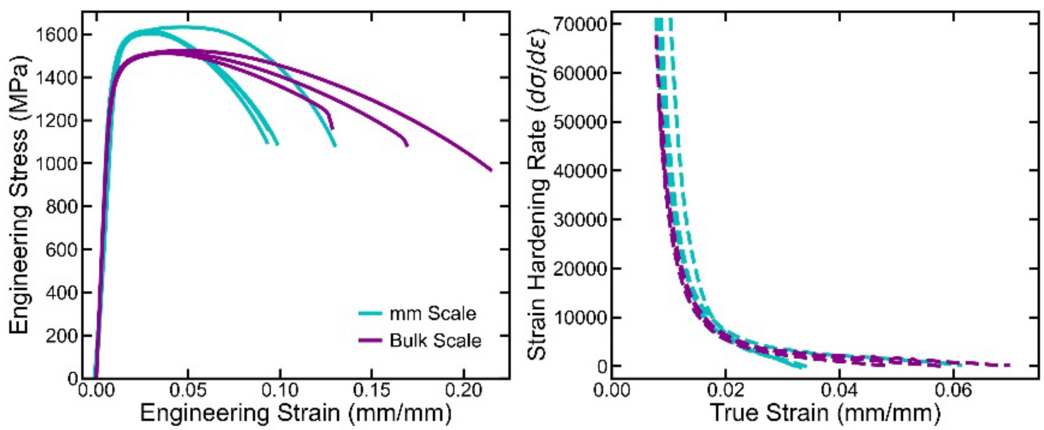


**Fig. 2 Summary of mesoscale and macroscale stress–strain curves**

The average tensile properties are summarized in Table 1. The bulk tensile properties agree well with the typical properties of commercially produced HP-9-4-30.[23] Potential origins of the discrepancies in the tensile properties will be discussed in detail in the following sections.

**Table 1 Summary of average tensile properties for mesoscale and macroscale specimens**

| Specimen | Yield strength (MPa) | Tensile strength (MPa) | Uniform elongation | Total elongation |
|---|---|---|---|---|
| Mesoscale | 1439 ±35 | 1616 ±12 | 0.034 ±0.009 | 0.105 ±0.018 |
| Macroscale | 1321 ±13 | 1517 ±6 | 0.041 ±0.005 | 0.171 ±0.043 |

## 4. Discussion

The comparison of mesoscale and macroscale tension tests indicates that the overall behavior of the material can be captured using miniaturized testing strategies. However, measurements of key material property descriptors (i.e., yield strength, tensile strength, and elongation) deviate from bulk measurements when reducing the specimen size. The origins of such discrepancies and potential mitigation will be discussed here.

## 4.1 Notch Strengthening

It is notable that the tensile strength is greater in the mesoscale specimens as compared to the macroscale samples. While the mesoscale specimens are small compared to the macroscale sample, they are not so small as to enter the regime of "smaller is stronger" commonly explored at the microscale. Instead, the increased tensile strength is hypothesized to be a result of notch strengthening.

Notch strengthening is a phenomenon where the apparent strength of a notched specimen is greater than that of a smooth specimen. It is characterized by the notch-strength ratio (NSR) according to

$$NSR = \frac{S_{NET}}{S_{UTS}}\,, \tag{1}$$

where $S_{NET}$ is the nominal tensile strength of a notched specimen and $S_{UTS}$ is the tensile strength of a smooth specimen.[24]

A materials capacity for notch strengthening depends on both material properties and the notch geometry. In general, a high post-necking ductility, as characterized by the ratio of post-necking strain to total elongation ($\delta_l/\delta_t$), and high stress triaxiality will lead to a high NSR.[25] High post-necking ductility indicates the ability to accommodate localized deformation while high stress triaxiality increases the incremental load for a given unit of plastic strain. Here, the macroscale tensile behavior reveals a significant amount of post-necking ductility; an average $\delta_l/\delta_t$ of 0.76 was observed. While no specific notch or flaw was observed, these results indicate that the material should exhibit a high degree of notch strengthening.

For future testing, it is important to consider the potential origins of notches and stress concentrators. It is assumed that they originate from machining processes used to fabricate specimens. The mesoscale samples were end-mill machined on the sides and could not be polished to remove defects in the form of notches generated from the chip. Such stress concentrators may arise at the transition from the gage section to the grips or due to chatter during machining. A representative example of a machining-induced notch is shown in Fig. 3. To eliminate such stress concentrators, alternative sample geometries and fabrication methods should be explored. For instance, increasing the radius at the transition to the grip should reduce stress concentrations. Alternative fabrication approaches may include electrical discharge machining (EDM) or laser micromachining. These methods are discussed in more detail with respect to surface damage layers in Section 4.3.

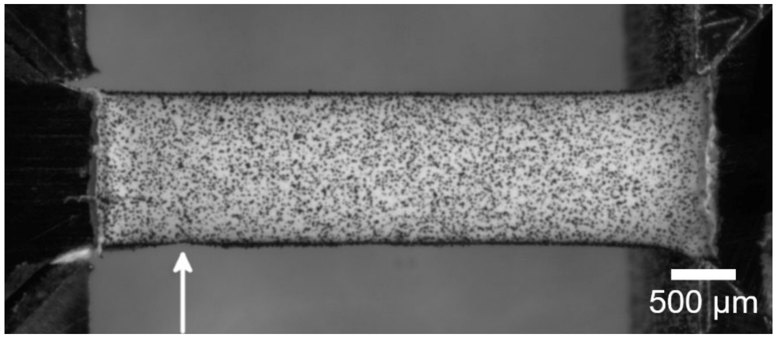


**Fig. 3 A representative example of a notch, as indicated by the arrow, originating from the machining process**

## 4.2 Slimness Ratio

The reduced total elongation in the mesoscale tension specimens can in part be attributed to the length of the gage section relative to cross-sectional area. This effect is known as the Slimness Ratio,[26] defined as

$$K = \frac{L}{\sqrt{A}}, \tag{2}$$

where $K$ is the slimness ratio, $L$ is the gage length, and $A$ is the cross-sectional area. The total elongation is related to the slimness ratio according to

$$e = e_0 K^{-a}, \tag{3}$$

where $e$ is the elongation and $e_0$ and $a$ are material-specific constants. The average $K$ for the mesoscale specimens was 5.67 while the macroscale specimens average 3.41. As a result, the mesoscale specimens would be expected to exhibit a lower overall strain level as compared to the macroscale specimens, which would be logical because the alloys and heat treatments were identical making $e_0$ and $a$ the same. However, the slimness ratios for all specimens are close to those suggested in many testing standards. If it is necessary to obtain comparable elongation values across length scales, care should be taken to ensure similar slimness ratios are employed.

## 4.3 Other Testing Challenges

Several additional systematic challenges in miniaturized tension testing were identified. These challenges pertain to both specimen fabrication and testing. Namely, the potential for hardening the specimen surface and issues with sample alignment may influence the measured tensile response.

Metal machining processes are well known to introduce surface damage layers whose thickness varies with the method employed. For instance, in steel, damage can extend to depths greater than 100 μm into a part depending on the cutting method and steel grade.[27] Such surface layers exhibit increased hardness due to work hardening and thermal effects. While these are typically disregarded in bulk material fabrication, for the mesoscale geometry employed here, the affected surface layers represent greater than 20% of the total cross-sectional area. In contrast, for conventionally machined bulk tensile specimens, the affected surface layer only comprises approximately 5% of the total cross section.

However, surface damage layers are readily mitigated through a variety of potential fabrication techniques. For instance, optimizing the milling parameters or using EDM can greatly reduce the thickness of a surface damaged layer as well as the induced residual stresses.[27] EDM may also introduce surface effects such as a recast layer and residual stresses. However, proper finishing strategies minimized this effect[27] and EDM was successfully employed in miniature tension tests.[28] An alternative fabrication approach is to use laser micromachining methods. In particular, femtosecond laser machining has been demonstrated as an effective method to produce tensile specimens[29] while minimizing subsurface damage to less than 1 μm.[30]

Finally, sample alignment plays an important role in the measured tensile response of mesoscale specimens. Both in-plane and out-of-plane misalignments can introduce artifacts into the tensile behavior. Both misalignments will introduce bending stresses within the gage section. For ductile materials, including most metals, bending stresses are of limited impact. However, for brittle materials, such as ceramics, bending stresses can lead to premature fracture and underestimation of the fracture strength. As such, extreme care must be taken to align the tensile grips. In-plane misalignment can be further ameliorated by changing the specimen geometry to a round grip shoulder (Fig. 4). By adopting a rounded shoulder, the specimen is better able to self-align within the grips, thereby minimizing misalignments.

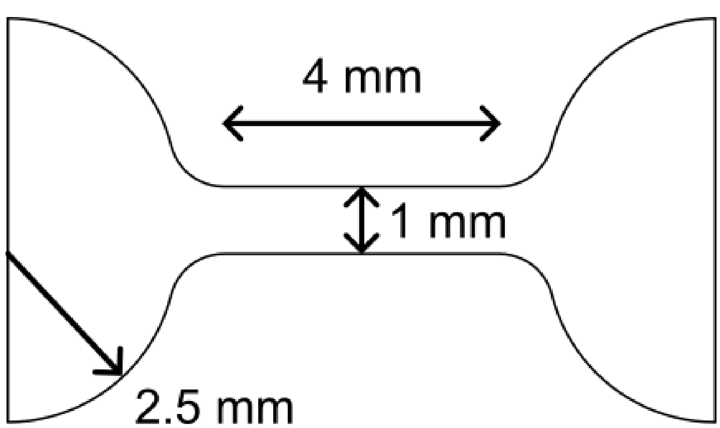


**Fig. 4 Alternative grip geometry to minimize in-plane misalignment**

Sample alignment may also introduce artifacts into the stress–strain curve such as initial nonlinearities at low loads. Out-of-plane misalignments can result in out-of-plane bending and erroneous strain measurements at low strains. As a result, mesoscale stress–strain curves often require a foot correction to adjust the point of zero strain.[26] Initial nonlinearities also impact the determination of the yield point by obscuring the linear elastic portion of the stress–strain response, thereby introducing uncertainty into the determination of the yield strength. To mitigate these effects, an initial preloading step may be introduced. By applying a sufficiently high load within the elastic limit, the specimen may be settled within the grips resulting in better alignment.

## 5. Conclusion

The tensile properties of a high-strength HP 9-4-30 steel were evaluated using macroscale and mesoscale specimen geometries to facilitate the direct comparison of the measured properties. The tensile response at both the meso- and macroscale was found to be comparable. However, the extracted tensile properties were found to depend on specimen length scale. In general, the mesoscale specimens exhibited higher strengths and lower ductility.

The discrepancies in measured tensile properties were assessed with regards to extrinsic effects arising from sample fabrication and testing. Differences in tensile strength were attributed to notch strengthening, where stress concentrators from the fabrication process artificially increase the tensile strength. The reduced elongation in the mesoscale samples stemmed from a combination of the sample geometry, as characterized by the slimness ratio, and stress concentrators promoting early necking. Finally, the increased tensile strength in the mesoscale samples was suggested to be a result of surface damaged layers produced during mechanical milling.

Potential mitigation strategies to alleviate these extrinsic effects were also described. These strategies focus on alternative fabrication methods and sample geometries. Transitioning to laser-based machining methods is anticipated to significantly reduce the influence of surface damage layers and provide greater precision during machining to minimize notching. Modifications to the specimen geometry will also allow comparable slimness ratios between macroscale and mesoscale tests, reduce stress concentrations, and improve self-alignment of specimens within the load frame.

Overall, this study and the proposed updates to the test methodology demonstrates that mesoscale tension testing will be an effective tool for future high-throughput screening of material properties.

## 6. References


1. LaVan DA, Sharpe WN. Tensile testing of microsamples. Exp Mech. 1999;39(3):210–216.

2. Sharpe WN. Murray lecture tensile testing at the micrometer scale: opportunities in experimental mechanics. Exp Mech. 2003;43(3):228–237.

3. Oliver WC, Pharr GM. Measurement of hardness and elastic modulus by instrumented indentation: advances in understanding and refinements to methodology. J Mater Res. 2004;19:3–20.

4. Hemker KJ, Sharpe WN. Microscale characterization of mechanical properties. Annu Rev Mater Res. 2007;37:93–126.

5. Sebastiani M, Johanns KE, Herbert EG, Pharr GM. Measurement of fracture toughness by nanoindentation methods: recent advances and future challenges. Curr Opin Solid St Mater Sci. 2015;19(6):324–333.

6. Ast J, Ghidelli M, Durst K, Göken M, Sebastiani M, Korsunsky AM. A review of experimental approaches to fracture toughness evaluation at the micro-scale. Mater Des. 2019;173:107762.

7. Uchic MD, Dimiduk DM, Florando JN, Nix WD. Sample dimensions influence strength and crystal plasticity. Science. 2004;305(5686):986–989.

8. Greer JR, De Hosson JTM. Plasticity in small-sized metallic systems: intrinsic versus extrinsic size effect. Prog Mater Sci. 2011;56(6):654–724.

9. Lee S-W, Nix WD. Size dependence of the yield strength of fcc and bcc metallic micropillars with diameters of a few micrometers. Philos Mag. 2012;92(10):1238–1260.

10. Hemker KJ, Last H. Microsample tensile testing of LIGA nickel for MEMS applications. Mater Sci Eng: A. 2001;319–321:882–886.

11. Sharpe WN, Turner KT, Edwards RL. Tensile testing of polysilicon. Exp Mech. 1999;39(3):162–170.

12. Magagnosc DJ, Schuster BE. Fracture strength of hot-pressed silicon carbide at the microscale. Mater Sci Eng: A. 2019;765:138297.

13. Ast J, Matthey B, Herre P, Höhn S, Herrmann M, Christiansen SH. Micro-cantilever testing of diamond – silicon carbide interfaces in silicon carbide bonded diamond materials produced by reactive silicon infiltration. Open Ceram. 2021;8:100176.

14. Hautier G. Finding the needle in the haystack: materials discovery and design through computational ab initio high-throughput screening. Comput Mater Sci. 2019;163:108–116.

15. Curtarolo S, Hart GL, Nardelli MB, Mingo N, Sanvito S, Levy O. The high-throughput highway to computational materials design. Nat Mater. 2013;12(3):191–201.

16. Hastings T, Mulukutla M, Khatamsaz D, Salas D, Xu W, Lewis D, Person N, Skokan M, Miller B, Paramore J, et al. An interoperable multi-objective batch Bayesian optimization framework for high-throughput materials discovery. arXiv preprint arXiv:2405.08900. 2024.

17. Kaufmann K, Vecchio KS. Searching for high entropy alloys: a machine learning approach. Acta Materialia. 2020;198:178–222.

18. Gianola DS, della Ventura NM, Balbus GH, Ziemke P, Echlin MP, Begley MR. Advances and opportunities in high-throughput small-scale mechanical testing. Curr Opin Solid St Mater Sci. 2023;27(4).

19. Heckman NM, Ivanoff TA, Roach AM, Jared BH, Tung DJ, Brown-Shaklee HJ, Huber T, Saiz DJ, Koepke JR, Rodelas JM, Madison JD, Salzbrenner BC, Swiler LP, Jones RE, Boyce BL. Automated high-throughput tensile testing reveals stochastic process parameter sensitivity. Mater Sci Eng: A. 2020;772.

20. Lu C, Danzer R, Fischer FD. Scaling of fracture strength in ZnO: effects of pore/grain-size interaction and porosity. J Eur Ceram Soc. 2004;24(14):3643–3651.

21. Echlin MP, Lenthe WC, Pollock TM. Three-dimensional sampling of material structure for property modeling and design. Integr Mater Manuf Innov. 2014;3(1):278–291.

22. ASTM E8-22. Standard test methods for tension testing of metallic materials. ASTM International; 2022.

23. Philip TV, McCaffrey TJ. Ultrahigh-strength steels. Properties and selection: irons, steels, and high-performance alloys. ASM Handbook, vol 1. 1990;430–448.

24. Dieter GE. Mechanical behavior under tensile and compressive loads. In: Kuhn H, Medlin D, editors. Mechanical testing and evaluation. ASM Handbook, vol 8. 2000;99–108.

25. Majima T, Anzai M, Nakazawa H. Notch tensile strength of ductile materials. Bulletin of JSME. 1986;29(258):4000–4004.

26. Holt JMT. Uniaxial tension testing. In: Kuhn H, Medlin D, editors. Mechanical testing and evaluation. ASM Handbook, vol 8. 2000;124–142.

27. Jawahir IS, Brinksmeier E, M'Saoubi R, Aspinwall DK, Outeiro JC, Meyer D, Umbrello D, Jayal AD. Surface integrity in material removal processes: recent advances. CIRP Annals. 2011;60(2):603-626.

28. Gussev MN, Howard RH, Terrani KA, Field KG. Sub-size tensile specimen design for in-reactor irradiation and post-irradiation testing. Nucl Eng Des. 2017;320:298–308.

29. Magagnosc DJ, Ligda JP, Sano T, Schuster BE. Femtosecond laser machining of micro-tensile specimens for high throughput mechanical testing. In: Starman L, Hay J, editors. Micro and Nanomechanics, Vol 5. Springer International Publishing; 2018:7–9.

30. Echlin MP, Titus MS, Straw M, Gumbsch P, Pollock TM. Materials response to glancing incidence femtosecond laser ablation. Acta Materialia. 2017;124:37–46.

## List of Symbols, Abbreviations, and Acronyms

| | |
|---|---|
| 3D | three-dimensional |
| DIC | digital image correlation |
| EDM | electrical discharge machining |
| NSR | notch-strength ratio |