

Nanoscale Architecture Unifies Electron and Dislocation Transport in Metals

A. K. Giri

United States Army Combat Capabilities Development Command

M. Martinelli

University of Kentucky

B. C. Hornbuckle

United States Army Combat Capabilities Development Command

A. J. Roberts

United States Army Combat Capabilities Development Command

M. Weisenberger

University of Kentucky

K. A. Darling

`kristopher.a.darling.civ@army.mil`

United States Army Combat Capabilities Development Command

Article

Keywords:

Posted Date: October 22nd, 2026

DOI: <https://doi.org/10.21203/rs.3.rs-11223919/v1>

License:   This work is licensed under a Creative Commons Attribution 4.0 International License.
[Read Full License](#)

Additional Declarations: No competing interests reported.

Nanoscale Architecture Unifies Electron and Dislocation Transport in Metals

Authors

A.K. Giri¹, M. Martinelli², B.C. Hornbuckle¹, A.J. Roberts¹, M. Weisenberger², and K.A. Darling^{1*}

Affiliations

¹Army Research Directorate, DEVCOM Army Research Laboratory, Aberdeen Proving Ground, MD, 21005, USA.

²Center for Applied Energy Research, University of Kentucky, 2540 Research Park Drive, Lexington, KY, 40511, USA.

*Corresponding author. Email: kristopher.a.darling.civ@army.mil (K.A.D.)

Abstract

Thermal transport and plastic deformation in metals are usually treated as separate processes. Here, we show that a stable nanoscale architecture can place a common limit on both. Using a bulk nanostructured Cu–Ta alloy, we identify a dense network of interfaces spaced about 15 nm apart that constrains the motion of electrons and dislocations. This architecture produces a broad thermal-conductivity plateau from about 175° to 375°C, with temperature-dependent behavior returning only when the intrinsic electron transport length becomes comparable to the interface spacing. The same nanoscale spacing also interrupts dislocation motion before high-velocity behavior can develop. Because the Cu–Ta structure remains stable across temperature and loading conditions, these effects can be directly linked to a single persistent structural length scale. These results establish nanoscale architecture as a common design parameter for controlling thermal and mechanical behavior in bulk metals.

Introduction

Microstructural architecture in materials is traditionally viewed as a passive modifier of physical behavior, influencing processes such as thermal transport and plastic deformation through independent scattering or interaction mechanisms^{1,2}. Within this conventional framework, these processes are treated as fundamentally distinct, governed by different carriers and physical descriptions. Electrons transport heat through the material, while dislocations move through the crystal to enable plastic deformation, with internal interfaces acting as localized perturbations that modify their motion through additive contributions to scattering rates or drag forces^{1,2}.

For plastic deformation, the Orowan relation ($\dot{\epsilon} = \rho b v$) describes the rate of plastic strain ($\dot{\epsilon}$) in terms of dislocation density (ρ), Burgers vector (b), and velocity (v)^{3,4}. In parallel, thermal transport by electrons and phonons is governed by carrier velocities and characteristic transport lengths (1). Although these processes are typically treated within separate physical frameworks, both are fundamentally carrier-flux processes governed by the density of mobile carriers, their velocity, and the physical quantity transported per carrier (see Supplementary Table 1 in Supplementary Information).

This description implicitly assumes that the characteristic transport or motion lengths of these carriers remain large compared to internal microstructural dimensions, such that interfaces modify carrier behavior without imposing fundamental constraints on transport. However, when internal architectural length scales approach or fall below these intrinsic length scales, this assumption breaks down. In this regime, the architecture can cease to act simply as a secondary perturbation and instead place an upper bound on the distance available for carrier motion. At this point, the architecture can determine not only how strongly a carrier is scattered or impeded, but whether that carrier can reach the transport regime it would normally access in an unconstrained material. This breakdown gives rise to a wide range of architecture-controlled transport phenomena. Quantum confinement represents one well-known example of transport behavior emerging when dimensions become sufficiently small⁵⁻⁷. Although the processes considered here are not quantum-confined, the broader length-scale principle is similar: sufficiently small dimensions can change the governing physics of transport rather than simply perturb it. The same underlying principle extends more generally to any transport process in which internal structural dimensions become comparable to the characteristic motion lengths of the governing carriers, driving a transition from temperature- or mechanism-controlled behavior to architecture-limited transport⁵⁻¹⁰.

This naturally raises a broader question: can a single nanoscale architecture simultaneously control the transport of electrons and dislocations within a unified physical framework? Addressing this question requires experimental systems in which the internal structural spacing is of relevant scale, well defined, and stable. Only under these conditions do carrier-interface interactions become sufficiently prevalent to dominate transport. A material architecture satisfying these criteria provides a platform for studying multiple carrier populations within the same

structural framework, allowing their transport behavior to be compared without complications from microstructural evolution.

To date, experimental systems satisfying these requirements have been largely limited to thin films, nanowires, and nanoparticles, where nanoscale dimensions inherently constrain carrier motion¹¹⁻¹³. Length scales capable of imposing this type of constraint are far less common throughout the three-dimensional interior of fully dense bulk metals. Such confinement is readily achieved when the physical dimensions of a material are reduced to the nanoscale, but reproducing the same constraint internally throughout a macroscopic solid is far more difficult. Achieving comparable behavior in bulk, three-dimensional nanostructured metals has remained a longstanding challenge because producing fully dense, thermally stable nanostructured microstructures has historically been difficult. Consequently, most studies have been limited to low-dimensional systems, which do not capture the role of a three-dimensional internal interface network in governing transport within bulk materials.

Recent advances in thermally stable bulk nanocrystalline alloys, particularly in Cu–Ta systems, provide a unique platform to address this challenge¹⁴. In these materials, grain size and nanoscale solute cluster distributions remain stable over extended temperature ranges, establishing a fixed architectural length scale within a bulk, fully dense system¹⁴. This enables direct interrogation of transport behavior in a regime where intrinsic carrier mean free paths or motion lengths are comparable to, or exceed, internal structural dimensions. Under these conditions, the architecture can become the limiting length scale itself, potentially preventing carriers from reaching the behavior that would otherwise emerge from their intrinsic scattering or drag mechanisms.

In this work, we report thermal conductivity measurements of nanocrystalline Cu–3 at.% Ta (Cu–3Ta) from room temperature to 600°C (64% of its absolute melting temperature) and demonstrate that heat transport is governed by a characteristic nanoscale architectural length scale rather than by temperature-dependent scattering over most of the measured range. By correlating thermal transport behavior with nanoscale cluster spacing measured by atom probe tomography and grain structure measured by scanning transmission electron microscopy, we identify a defect-limited regime in which the effective electron mean free path is truncated by the internal architecture. Remarkably, the characteristic length scale governing this thermal-transport regime coincides with that associated with deviations in dislocation glide under mechanical loading. At this length scale, dislocations are interrupted before they can accelerate into the ballistic, phonon-drag-dominated regime that would otherwise emerge at sufficiently high loading rates. Thus, the architecture does more than independently scatter electrons and obstruct dislocations. It prevents two fundamentally different carrier systems from accessing transport regimes that would otherwise naturally emerge. This correspondence reveals a previously unrecognized connection between electron and dislocation transport and establishes a unified framework in which microstructural architecture acts not as a perturbation, but as a common physical ceiling on carrier motion and a primary governing parameter for both thermal transport and deformation in nanostructured metals.

Results

Critically, the ability to interrogate transport over such a broad temperature range is made possible by the exceptional microstructural stability of the Cu–Ta system. In conventional nanostructured metals, elevated-temperature measurements are often accompanied by recovery, grain growth, solute redistribution, or precipitate coarsening, making it difficult to distinguish intrinsic changes in carrier transport from those arising from concurrent microstructural evolution. The stabilized Cu–Ta architecture largely removes this ambiguity. Previous studies of the Cu–Ta system^{15,16} reported negligible changes in grain size and nanocluster morphology following annealing at 800 °C, approximately 200 °C above the maximum temperature investigated here, for durations ranging from 1,000 to 10,000 h. The previously established thermal stability of Cu–Ta indicates that changes in grain size and Ta-rich cluster spacing should be negligible over the comparatively short duration of the present measurements, providing a basis for treating the characteristic internal architectural length scale as approximately constant across this temperature range. Consequently, changes in thermal conductivity can be attributed to evolving carrier–interface interactions rather than to concurrent evolution of the microstructure. This stability therefore provides a rare experimental platform for directly interrogating elevated-temperature physical transport within a bulk nanostructured metal.

With this exceptional microstructural stability established, we next identify and quantify the internal architecture that defines the characteristic length scale relevant to transport and deformation in the present material condition. Scanning transmission electron microscopy confirms that bulk Cu–3Ta retains a mean grain size of approximately 250 nm (Figure 1). High-angle annular bright and dark-field imaging reveals a stochastic distribution of Ta-rich clusters located both along grain boundaries and within grain interiors, forming a dense three-dimensional network of internal interfaces (Figure 1B-C).

Although occasionally larger Ta-rich particles (~50 nm in diameter) are observed, their number fraction is low, and they do not define the dominant structural length scale. Instead, the architecture is characterized by a pervasive nanoscale distribution of clusters that establishes a well-defined internal spacing. This stability and persistence of nanoscale features are consistent with prior detailed observations of exceptional resistance to grain growth and defect accumulation in Cu–Ta alloys¹⁷.

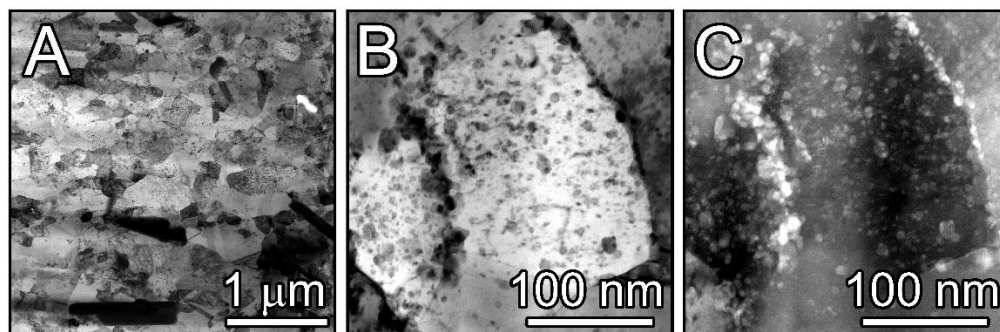


Figure 1. Microstructure of stabilized nanocrystalline Cu–3Ta. (A) Low-magnification STEM BF image showing a refined grain structure. (B) Higher magnification STEM BF revealing a dense distribution of Ta-rich nanoclusters within the grains. (C) High-resolution STEM HAADF image highlighting the nanoscale cluster network that defines the characteristic architectural length scale.

Atom probe tomography (Figure 2) further resolves this architecture by providing direct measurement of cluster size, chemistry, and spatial distribution. Ta-rich clusters exhibit diameters below 4 nm, consistent with coherent to semi-coherent interfaces with the Cu matrix¹⁷. Number density analysis yields cluster densities on the order of $3.26 \times 10^{23} \text{ m}^{-3}$, corresponding to an average intercluster spacing of approximately 14.6 nm which is taken as the characteristic length scale of the alloy. Complementary atom maps reveal the full three-dimensional distribution of Cu and TaO species, with oxygen isoconcentration surfaces (3 at. % O). TaO atom maps further confirm that these clusters are chemically complex, incorporating oxygen within the Ta-rich regions. Together, these measurements establish an average internal spacing of approximately 14.6 nm as the characteristic nanoscale length scale of the alloy. The spatial distribution of these features, together with the absence of a dominant coarse precipitate population, indicates that this length scale is defined by the nanoscale interface network rather than by grain size or larger isolated second-phase particles. This characteristic spacing provides the architectural length scale used in the following sections to analyze transport behavior.

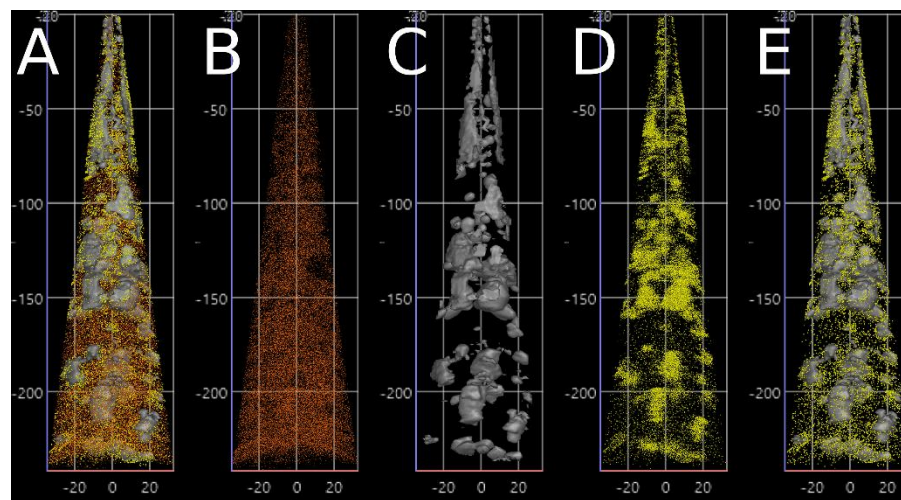


Figure 2. Atom Probe Tomography (A) Atom map for Cu–3Ta after 1 hour at 900°C displaying both Cu and TaO atoms as well as O isoconcentration surfaces set at 3 at. % O for an entire tip. (B) Cu atom map. (C) 3 at. % O isoconcentration surface present throughout the entire tip. (D) TaO atom map. (E) TaO atom map as well 3 at. % O isoconcentration surface displayed.

With the nanoscale architecture and its characteristic length scale established, we next examine how this internal interface network governs thermal transport behavior (Figure 3). In this context, thermal conductivity serves not only as a measurable property, but as a direct experimental signature of a broader architecture-limited transport regime that will also extend to dislocation motion.

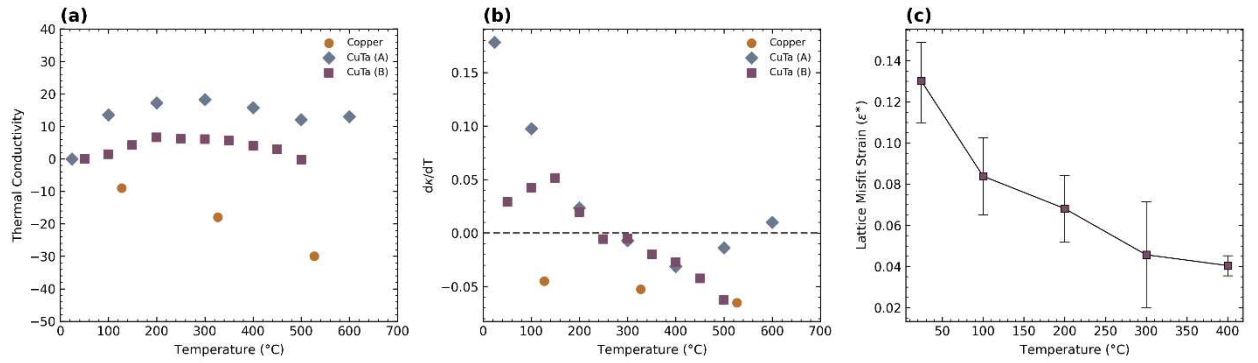


Figure 3: Temperature-dependent transport in Cu-3Ta compared to Cu. (A) Normalized thermal conductivity i.e. $(\kappa - \kappa_{RT})$ vs. temperature showing non-monotonic behavior and a plateau in Cu-3Ta. (B) Corresponding $d\kappa/dT$ highlighting a transition in transport regime. (C) Decreasing lattice misfit strain, consistent with interfacial relaxation, for Ta based clusters.

The thermal conductivity of nanostructured Cu-3Ta at room temperature was measured to be approximately $250\text{--}260\text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, substantially lower than that of coarse-grained pure Cu ($\approx 390\text{--}400\text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$), but comparable to that of pure Al ($237\text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$)^{1,18}. This reduced thermal conductivity reflects the imposition of the network of Ta-rich clusters, which constrains the effective electron mean free path to the architectural length scale identified above^{19,20}. As shown in Figure 3A, this manifests not only as a reduced absolute conductivity relative to Cu, but also as a fundamentally different temperature dependence, with Cu-3Ta deviating from the monotonic decline characteristic of pure metals.

More specifically, from $24\text{ }^{\circ}\text{C}$ to $175\text{ }^{\circ}\text{C}$, Figure 3A reveals that both thermal conductivity measurements of Cu-3Ta increase modestly, whereas pure Cu exhibits a linear decrease. Such a decrease is the conventional behavior of metals and metallic alloys, arising primarily from increasing electron-phonon scattering as temperature rises. Cu-3Ta therefore exhibits a clear departure from conventional metallic behavior, reflected in the reversal of the temperature dependence of thermal conductivity. As shown in Figure 3B, $d\kappa/dT$ is positive for Cu-3Ta over this regime, whereas it remains negative for pure Cu. Because the nanoscale architecture remains effectively fixed over this temperature range, this positive slope indicates that a temperature-dependent source of transport resistance must be decreasing sufficiently to offset the expected increase in electron-phonon scattering. This behavior is therefore consistent with reduced electron scattering or improved carrier transmission across the internal interfaces, leading to a modest decrease in overall transport resistance while the governing architectural length scale remains unchanged, as examined below.

From $175\text{ }^{\circ}\text{C}$ to approximately $375\text{ }^{\circ}\text{C}$, this evolution gives way to a broad thermal-conductivity plateau with minimal temperature dependence (Figure 3A), as $d\kappa/dT$ approaches zero (Figure 3B). The near-zero slope indicates that the net transport resistance has become effectively insensitive to temperature over this interval, consistent with the internal architecture imposing the dominant constraint on electron transport. Above approximately $400\text{ }^{\circ}\text{C}$, thermal conductivity begins to

decline and $d\kappa/dT$ becomes increasingly negative, signaling a transition toward the conventional temperature-dependent behavior associated with increasing electron–phonon scattering.

To quantify this crossover from architecture-limited to intrinsic electron–phonon-controlled transport, the temperature-dependent electron mean free path in pure Cu was estimated from published electrical-resistivity data using a Drude–Sommerfeld treatment. The calculated mean free path decreases from approximately 39 nm at room temperature to 23 nm at 200 °C, 18 nm at 300 °C, 15 nm at 400 °C, and 11 nm at 600 °C (see Supplementary Note 2 and Supplementary Tables 2 and 3 in Supplementary Information). Throughout most of the 175 to 375 °C thermal-conductivity plateau, the intrinsic electron mean free path remains larger than the measured intercluster spacing of approximately 14.6 nm in Cu–3Ta:

$$\ell_e > L_{\text{arch}},$$

Under this condition, the shorter architectural length scale limits the effective distance available for electron transport, such that the internal interface network, rather than the intrinsic electron–phonon scattering length, provides the dominant transport constraint. Near 400 °C, the two length scales become comparable:

$$\ell_e \approx L_{\text{arch}}$$

This marks the crossover to a regime in which intrinsic electron–phonon scattering increasingly contributes to the decline in thermal conductivity. At still higher temperatures:

$$\ell_e < L_{\text{arch}}$$

The intrinsic scattering length becomes shorter and increasingly becomes the dominant constraint on transport. To further understand the origin of the unexpected increase in thermal conductivity preceding the plateau, we draw on our earlier in situ high-temperature STEM strain measurements (Figure 3C), originally used to examine the temperature dependence of mechanical strength. These measurements showed a progressive reduction in interfacial misfit strain and increasing coherency between the Ta clusters and Cu matrix with increasing temperature, likely driven by differential thermal expansion between the two phases²¹. Reconsidered in the present context, these observations provide a physical basis for the temperature-dependent evolution of electron scattering across the fixed nanoscale architecture.

As the lattice mismatch decreases, electron scattering at the cluster interfaces is expected to decrease, allowing carrier transmission across the fixed interface network to become progressively more efficient. Notably, the majority of the interfacial misfit strain is relieved by approximately 175 °C, after which only minimal additional evolution is observed. This stabilization coincides with the onset of the thermal-conductivity plateau, supporting an interpretation in which the architecture remains fixed while the scattering efficiency of its interfaces evolves at lower temperatures and subsequently approaches a steady-state configuration.

Together, these results establish that heat conduction in Cu–3Ta operates within an architecture-limited transport regime over a substantial temperature range. The effective electron transport length is capped by the nanoscale interface spacing while the intrinsic electron mean free path remains larger than this architectural length scale, with conventional electron–phonon scattering becoming increasingly important only once the two length scales approach and cross near 400 °C.

The architectural constraint identified here for electron transport corresponds to the same nanoscale length scale previously shown to govern dislocation motion under dynamic loading. Reconsidered in the present context, these earlier observations provide a basis for linking plastic deformation and thermal transport within a common framework in which carrier motion is limited by an intrinsic structural length scale.

The current authors previously established a dislocation framework in which the accessible dislocation velocity is governed by the distance required to accelerate into the ballistic regime where phonon drag becomes operative. Figure 4A presents the normalized dislocation velocity (v/v_s) as a function of travel distance in Cu, calculated using the Gillis–Kratochvil formulation^{22,23} for dislocation acceleration under an applied resolved shear stress with a drag force opposing motion (see Supplementary Note 3 in Supplementary Information). In an unconstrained lattice, a dislocation requires approximately 85 nm of uninterrupted glide to approach a steady-state velocity of $\sim 0.6 v_s$, corresponding to the onset of strong dislocation–phonon interactions (see Figure 4A).

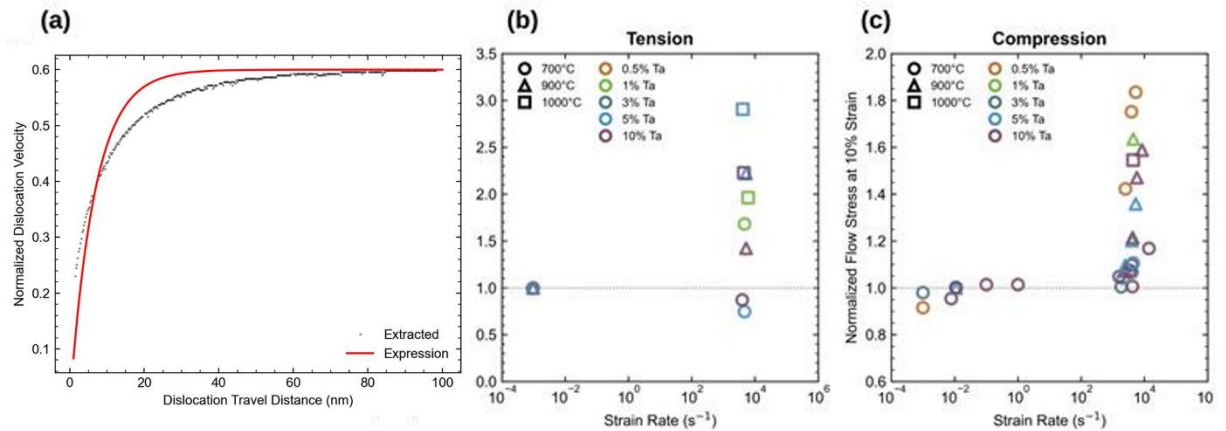


Figure 4: Dislocation transport and strain-rate-dependent mechanical response. (A) Normalized dislocation velocity as a function of travel distance, showing rapid saturation toward a limiting velocity. (B) Normalized flow stress in tension as a function of strain rate for varying Ta concentrations and temperatures. (C) Corresponding compression data, showing increased strain-rate sensitivity with increasing cluster spacing (lower Ta content and/or higher processing temperature).

In the present case, however, the measured Ta nanocluster spacing of approximately 15 nm, together with the smaller spacings present in related Cu–Ta microstructures, falls well below the intrinsic acceleration length required for dislocations to reach the ballistic regime. Consequently, the distance available for

uninterrupted glide between successive interfaces is insufficient for a dislocation to accelerate to the velocities at which phonon drag becomes dominant. Dislocation motion is therefore repeatedly interrupted by the internal interface network before the ballistic regime can be reached. The attainable dislocation velocity is thus bounded by the same architectural length scale that constrains electron transport, preventing access to the phonon-drag-dominated regime²³.

Experimental support for this architecture-limited dislocation behavior can be identified through the strain-rate dependence of flow stress. In an unconstrained material, increasing strain rate allows dislocations to access progressively higher velocities, eventually producing a characteristic upturn in flow stress as phonon drag becomes increasingly important. If the available glide distance is instead truncated by a dense nanoscale interface network, this high-rate upturn should be suppressed or dampened, providing an experimental fingerprint of architecture-limited dislocation transport.

The Cu–Ta alloy series examined previously provides a means of testing this behavior because changes in Ta concentration and processing temperature systematically alter the Ta nanocluster population and, consequently, the characteristic intercluster spacing. Higher Ta concentrations and lower processing temperatures preserve a high number density of Ta-rich clusters and shorter intercluster spacings, whereas decreasing the Ta concentration or increasing the processing temperature reduces the cluster density through coarsening and increases the distance available for dislocation motion¹⁵.

The reported strain-rate-dependent flow-stress measurements from the current authors (Figure 4B–C), obtained at 0.2% strain in tension and 10% strain in compression²⁴, follow this expected trend. Alloys containing the highest densities of Ta-rich clusters exhibit only minimal increases in normalized flow stress with increasing strain rate, whereas conditions with lower cluster densities and larger spacings show a progressively stronger high-rate response. Importantly, suppression of the flow-stress upturn occurs in microstructures where the intercluster spacing is comparable to, or smaller than, the critical acceleration length. Under these conditions, dislocations encounter successive interfaces before they can attain the velocities associated with the phonon-drag-dominated regime, giving rise to architecture-limited dislocation transport. Reconsidered in the present context, these measurements therefore provide an experimental basis for the dislocation-transport constraint predicted in Figure 4A and its connection to the architectural limitation on electron transport identified here.

Beyond the architecture-limited electron and dislocation transport identified here, the nanoscale microstructure also has important consequences for lattice dynamics and can reshape the phonon spectrum through the presence of Ta-rich clusters, their associated interfaces, and grain boundaries^{23–25}. The dense interfacial network introduces local variations in atomic mass, interatomic bonding, and lattice strain, which collectively alter the phonon density of states relative to that of pure Cu. These nanoscale heterogeneities can give rise to localized vibrational modes, broadened phonon distributions, and modified group velocities, effectively redistributing phonon states²⁶. These changes represent more than additional scattering pathways and instead reflect a fundamental modification of the

vibrational environment imposed by the internal architecture. This redistribution is important because it modifies the interactions of phonons with both dislocations and electrons, providing an additional pathway through which the same nanoscale architecture influences the two transport processes considered here.

For dislocations, this altered phonon population directly affects phonon drag²⁷. In Cu–Ta, nanoscale compositional heterogeneity and the associated interfaces preferentially scatter higher-frequency phonons, producing a redistribution toward lower-frequency, long-wavelength modes^{23–25}. These modes couple more effectively to dislocation strain fields and therefore enhance phonon drag coefficient. Thus, the nanoscale architecture affects dislocation motion in two distinct ways. Redistribution of the phonon spectrum increases the dissipative resistance associated with dislocation–phonon coupling; however, the short interfacial spacing remains the dominant constraint, truncating glide before dislocations can accelerate into a phonon-drag-dominated regime.

In contrast, the same redistribution of the phonon density of states has the opposite effect on electron–phonon interactions. The reduction in high-frequency phonons decreases the population of modes most effective at scattering electrons, thereby reducing electron–phonon scattering and, in an unconstrained material, increasing the electron mean free path. However, the short interfacial spacing remains the dominant transport constraint, capping the effective electron transport length before this increased intrinsic mean free path can be realized. Thus, the phonon redistribution modifies the underlying scattering environment, but the nanoscale architecture ultimately determines the distance over which electrons can transport energy.

Conclusions

This work demonstrates that thermally stable nanostructured metals can sustain an architecture-limited transport regime across broad temperature and loading conditions, in which a persistent nanoscale architecture, rather than microstructural evolution or intrinsic carrier interactions alone, governs the accessible transport behavior. When the characteristic spacing of the internal interface network approaches or falls below the intrinsic transport lengths of electrons and dislocations, carrier motion becomes truncated by the architecture itself. Under these conditions, the internal structural length scale can determine whether a carrier is able to access the transport regime that would otherwise emerge from its intrinsic scattering or drag mechanisms. The exceptional microstructural stability of nanostructured Cu–Ta makes this behavior experimentally accessible by preserving the governing nanoscale architecture throughout the thermal and mechanical measurements.

These results further establish that thermal transport and plastic deformation need not be treated as fundamentally independent phenomena in thermally stable nanostructured materials. Although electrons and dislocations interact with the microstructure through different physical mechanisms, both can ultimately be constrained by the same internal structural length scale. The resulting behavior can therefore be understood within a common framework in which architecture places

an upper bound on carrier motion and determines the transport regimes that can be accessed.

Taken together, the phonon redistribution drives the two carrier systems in opposite directions, enhancing drag on dislocations while reducing electron–phonon scattering. Yet in both cases, the accessible transport regime remains set by the same nanoscale intercluster spacing. Thus, the phonon spectrum modulates carrier interactions, whereas the architecture defines the governing transport length.

More broadly, these findings establish microstructural architecture as a design parameter for controlling seemingly divergent transport processes within a bulk material. Rather than treating internal interfaces solely as passive scattering centers or barriers to motion, they can be viewed as structural elements that define the characteristic distances available for carrier transport. Engineering these length scales therefore provides a pathway for determining not only the magnitude of carrier interactions, but also which transport regimes can physically develop. This framework offers a general strategy for linking and tailoring thermal and mechanical behavior through a common nanoscale architecture.

Methods

Powder Production and Consolidation

Nanocrystalline Cu–3 at.% Ta (Cu–3Ta) powder used for the thermal-conductivity measurements was produced by low-temperature mechanical alloying in a Zoz mill for 14 h using an approximately 1 kg batch size²⁸. The mechanically alloyed powder was encapsulated and consolidated by hot isostatic pressing at 900 °C to produce fully dense billets. Following removal of the encapsulation can, the billets were hot extruded at 900 °C in a single pass, reducing the diameter from 3.5 to 1.15 in. Specimens for thermal-transport measurements were sectioned from the extruded billets, machined to the geometry required for laser-flash analysis, and mechanically polished. A separate series of Cu–Ta alloys was produced for the high-strain-rate tensile and compression studies. Five compositions, Cu–0.5 at. % Ta, Cu–1 at. % Ta, Cu–3 at. % Ta, Cu–5 at. % Ta, and Cu–10 at. % Ta were synthesized to distinguish the respective effects of grain boundaries and Ta-rich nanoclusters on strength, tensile ductility, strain-rate sensitivity, and the strain rate at which the flow-stress upturn occurs. The materials were processed by cryogenic mechanical alloying followed by equal-channel angular extrusion (ECAE) at 700, 900, or 1000 °C. The processing procedures followed those described previously in Refs.^{25, 29-31}. Elemental Cu and Ta powders were combined in the required proportions under an Ar atmosphere and loaded in 10 g batches into a 440C stainless-steel vial containing 440C stainless-steel milling media. Each batch was mechanically alloyed for 4 h while maintained at liquid-nitrogen temperature.

Following mechanical alloying, the as-milled powders were individually loaded into separate Ni cans that were subsequently sealed under Ar. The powder-filled cans were heated to the selected processing temperature and extruded through a 90° ECAE die to produce fully dense billets. Comparisons among compositions processed at the same temperature were used to examine the influence of Ta-rich nanoclusters while minimizing differences in grain size. Conversely, comparisons among materials processed at different temperatures were used to assess the combined influence of grain-size evolution and associated changes in the Ta-rich nanocluster population.

Microstructural Characterization

Microstructural characterization was performed on all material conditions used for both the thermal-transport and high-strain-rate mechanical studies using scanning transmission electron microscopy (STEM) and atom probe tomography (APT). TEM/STEM and APT specimens were prepared from the consolidated and extruded materials by focused ion beam lift-out. Electron microscopy was used to evaluate grain size, morphology, and the spatial distribution of Ta-rich features, while APT provided three-dimensional measurements of Ta-cluster size, number density, chemistry, and grain-boundary segregation. These analyses confirmed that materials produced by both processing routes retained the characteristic Cu–Ta microstructure, consisting of nanocrystalline Cu grains containing a dense distribution of nanoscale Ta-rich clusters and associated interfacial segregation. The characterization further verified that the nanoscale architectural features

governing thermal transport and high-rate deformation were intrinsic to the bulk material rather than specific to a single processing route or specimen geometry.

Thermal and Mechanical Property Measurements

Thermal diffusivity was measured using the laser-flash method in accordance with ASTM E1461³². Measurements were performed using Netzsch LFA 427 NanoFlash® and LFA 457 MicroFlash® systems located at the Army Research Laboratory and the University of Tennessee, Knoxville, respectively. Thermal conductivity was calculated from the measured thermal diffusivity, independently measured density, and heat capacity.

Because the heat capacities of most metals vary only weakly over a broad temperature range after the lattice vibrational modes become fully populated, the heat capacity of Cu–3Ta was estimated using a rule of mixtures. This approximation was further supported by the strongly immiscible nature of the Cu–Ta system. Cu and Ta remain largely phase separated, with negligible mutual solubility and no significant intermetallic compound formation. The alloy specific heat capacity was therefore approximated as the mass-fraction-weighted sum of the specific heat capacities of Cu and Ta.

Following consolidation, specimens for tensile and compression testing were machined parallel to the extrusion direction. Tensile specimens were prepared in a rectangular dog-bone geometry, while compression specimens were machined into cylinders measuring 3 mm in diameter and 3 mm in length. Quasi-static tensile and compression tests were performed over a range of strain rates using an electromechanical universal testing machine equipped with an extensometer. The measured engineering stress and strain data were converted to true stress and true strain.

High-strain-rate tensile and compression experiments were conducted using the Kolsky bar techniques described previously³³⁻³⁵. Specialized grips were used during tensile testing to accommodate the rectangular dog-bone specimens. Both the Kolsky bars and tensile grips were manufactured from Inconel 718. For tensile testing, a striker tube with mechanical impedance matched to that of the incident and transmitted bars impacted a flange attached to the incident bar, generating a tensile pulse that propagated toward the specimen. For compression testing, a striker bar impacted the incident bar and generated a compressive pulse that traveled toward the specimen positioned between the incident and transmitted bars.

In both configurations, interaction of the incident pulse with the specimen produced transmitted and reflected strain pulses. Strain gauges arranged in Wheatstone bridge configurations on the incident and transmitted bars were used to record these signals. The transmitted pulse provided a measure of the force carried through the specimen, while the reflected pulse was used to determine the specimen strain and strain rate^{24,34-36}.

The Kolsky bar experiments were conducted at strain rates on the order of 10^3 s^{-1} . Tensile specimens were deformed to failure, while the high-strain-rate compressive flow stress was evaluated at 10% strain. For the tensile experiments, the flow stress was evaluated at approximately 0.2% strain to compare the strain-

rate sensitivity of the different microstructures. Because force equilibrium may not be fully established at such a low strain during Kolsky bar testing, the tensile values were used as a qualitative measure of the relative increase in flow stress produced by high-rate deformation.

The tensile trends were evaluated together with the compression measurements, for which force equilibrium was achieved at higher strains. The Cu–Ta materials exhibited relatively limited strain hardening and an approximately elastic, perfectly plastic response. Consequently, the strain-rate-dependent trends evaluated at 0.2% strain in tension were consistent with those obtained at 10% strain in compression, supporting the use of the tensile measurements for comparative analysis among the different microstructural conditions.

High-strain-rate hardness was measured using laser-induced particle impact testing configured as a microparticle impact indentation experiment. Approximately 14 μm diameter spherical alumina particles were used as nominally rigid indenters and accelerated by laser ablation to velocities of several hundred meters per second before impacting polished Cu-3Ta target surfaces. Alumina was selected because its hardness is substantially greater than that of the metallic targets, allowing the plastic deformation to be localized primarily within the target material. Time-resolved optical imaging was used to track each particle immediately before and after impact, providing direct measurements of the impact velocity, v_i and rebound velocity, v_r . The kinetic energy dissipated during impact was then used to estimate the plastic work associated with formation of the residual indentation.

Following impact, the residual indentation geometry was reconstructed using laser confocal microscopy to determine the indentation depth and displaced volume. The dynamic hardness was calculated by dividing the plastic work by the measured indentation volume:

$$H_{\text{dynamic}} = \frac{W_{\text{plastic}}}{V_{\text{indent}}} = \frac{\frac{1}{2} m_p (v_i^2 - v_r^2)}{V_{\text{indent}}},$$

Where m_p is the microparticle mass. This approach combines real-time impact and rebound measurements with post-impact surface profilometry, enabling quantitative hardness measurements at characteristic strain rates above 10^6 s^{-1} ^{23,37}.

References

1. Kittel, C. Introduction to Solid State Physics 7th edn, p. 589 (Wiley, Singapore, 1996).
2. Hirth, J. P. & Lothe, J. Theory of Dislocations 2nd edn (Wiley, New York, 1982).
3. Orowan, E. Zur Kristallplastizität. III. Über den Mechanismus des Gleitvorganges. Z. Phys. 89, 634–659 (1934).
4. Hull, D. & Bacon, D. J. Introduction to Dislocations 5th edn (Butterworth-Heinemann, Oxford, 2011).
5. Brus, L. E. Electron–electron and electron–hole interactions in small semiconductor crystallites: the size dependence of the lowest excited electronic state. J. Chem. Phys. 80, 4403–4409 (1984).
6. Alivisatos, A. P. Semiconductor clusters, nanocrystals, and quantum dots. Science 271, 933–937 (1996).
7. García de Arquer, F. P. et al. Semiconductor quantum dots: technological progress and future challenges. Science 373, eaaz8541 (2021).
8. Carlton, C. E. & Ferreira, P. J. What is behind the inverse Hall–Petch effect in nanocrystalline materials? Acta Mater. 55, 3749–3756 (2007).
9. Naik, S. N. & Walley, S. M. The Hall–Petch and inverse Hall–Petch relations and the hardness of nanocrystalline metals. J. Mater. Sci. 55, 2661–2681 (2020).
10. Koch, C. C. & Narayan, J. The inverse Hall–Petch effect—fact or artifact? MRS Online Proc. Libr. 634, 511 (2000).
11. Ziman, J. M. Electrons and Phonons: The Theory of Transport Phenomena in Solids (Oxford Univ. Press, London, 1963).
12. Cahill, D. G. et al. Nanoscale thermal transport. J. Appl. Phys. 93, 793–818 (2003).
13. Cahill, D. G. et al. Nanoscale thermal transport. II. 2003–2012. Appl. Phys. Rev. 1, 011305 (2014).
14. Darling, K. A., Mishin, Y., Thadhani, N. N., Wei, Q. & Solanki, K. N. Mechanical behavior of microstructurally stable nanocrystalline alloys: processing, properties, performance, and prospects. Prog. Mater. Sci. 155, 101519 (2026).
15. Hornbuckle, B. C., Solanki, K. N. & Darling, K. A. Prolonged high-temperature exposure: tailoring nanocrystalline Cu–Ta alloys against grain growth. Mater. Sci. Eng. A 824, 141818 (2021).
16. Hornbuckle, B. C. et al. A high-temperature nanostructured Cu–Ta–Li alloy with complexion-stabilized precipitates. Science 387, 1413–1417 (2025).
17. Darling, K. A. et al. Extreme creep resistance in a microstructurally stable nanocrystalline alloy. Nature 537, 378–381 (2016).

18. Ho, C. Y., Powell, R. W. & Liley, P. E. Thermal conductivity of the elements. *J. Phys. Chem. Ref. Data* 1, 279–421 (1972).
19. Dong, H., Wen, B. & Melnik, R. Relative importance of grain boundaries and size effects in thermal conductivity of nanocrystalline materials. *Sci. Rep.* 4, 7037 (2014).
20. Islam, M. R. et al. Evaluating size effects on the thermal conductivity and electron–phonon scattering rates of copper thin films for experimental validation of Matthiessen’s rule. *Nat. Commun.* 15, 9167 (2024).
21. Rajagopalan, M. et al. Microstructural evolution in a nanocrystalline Cu–Ta alloy: a combined in situ TEM and atomistic study. *Mater. Des.* 113, 178–185 (2017).
22. Gillis, P. P. & Kratochvil, J. Dislocation acceleration. *Philos. Mag.* 21, 425–432 (1970).
23. Tang, Q. et al. Suppressed ballistic transport of dislocations at strain rates up to 10^9 s^{-1} in a stable nanocrystalline alloy. *Commun. Mater.* 6, 43 (2025).
24. Turnage, S. A. et al. Anomalous mechanical behavior of nanocrystalline binary alloys under extreme conditions. *Nat. Commun.* 9, 2699 (2018).
25. Srinivasan, S. et al. Role of tantalum concentration, processing temperature, and strain-rate on the mechanical behavior of copper–tantalum alloys. *Acta Mater.* 208, 116706 (2021).
26. Seyf, H. R. et al. Rethinking phonons: the issue of disorder. *npj Comput. Mater.* 3, 49 (2017).
27. Nabarro, F. R. N. *Theory of Crystal Dislocations* (Dover, New York, 1987).
28. Hammond, V. H. et al. Processing of bulk nanocrystalline metals at the US Army Research Laboratory. *J. Vis. Exp.* 133, e56950 (2018).
29. Segal, V. M. Materials processing by simple shear. *Mater. Sci. Eng. A* 197, 157–164 (1995).
30. Furukawa, M., Horita, Z., Nemoto, M. & Langdon, T. G. Review: processing of metals by equal-channel angular pressing. *J. Mater. Sci.* 36, 2835–2843 (2001).
31. Zhu, Y. T. & Lowe, T. C. Observations and issues on mechanisms of grain refinement during the ECAP process. *Mater. Sci. Eng. A* 291, 46–53 (2000).
32. ASTM International. ASTM E1461-13(2022), Standard Test Method for Thermal Diffusivity by the Flash Method (ASTM International, West Conshohocken, PA, 2022).
33. Kale, C. et al. On the roles of stress-triaxiality and strain-rate on the deformation behavior of AZ31 magnesium alloys. *Mater. Res. Lett.* 6, 152–158 (2018).
34. Kale, C. et al. Towards dynamic tension–compression asymmetry and relative deformation mechanisms in magnesium. *Materialia* 9, 100543 (2020).
35. Barton, D. J. et al. Microstructure and dynamic strain aging behavior in oxide-dispersion-strengthened 91Fe–8Ni–1Zr (at.%) alloy. *Mater. Sci. Eng. A* 725, 503–509 (2018).

- 681 36. Gama, B. A., Lopatnikov, S. L. & Gillespie, J. W. Jr. Hopkinson bar
682 experimental technique: a critical review. *Appl. Mech. Rev.* 57, 223–250
683 (2004).
- 684 37. Hassani, M., Veyssset, D., Nelson, K. A. & Schuh, C. A. Material hardness at
685 strain rates beyond 10^6 s^{-1} via high-velocity microparticle impact indentation.
686 *Scr. Mater.* 177, 198–202 (2020).
687
688

689 **Supplementary Information** is available in the online version of the paper.
690

691 **Acknowledgements**

692 This work was supported by the DEVCOM Army Research Laboratory.
693 Additionally, this work was performed under a cooperative agreement between the
694 DEVCOM Army Research Laboratory and University of Kentucky.
695
696

697 **Author contributions**

698 A.J.R., A.K.G., B.C.H., M.M. and M.W. conducted the experiments. K.A.D.,
699 B.C.H., M.M. and M.W. analyzed the data. K.A.D. conceptualized and designed
700 the project. A.K.G. and K.A.D. wrote the manuscript.
701

702 **Competing interests:** The authors declare they have no competing interests.
703
704

705 **Data availability**

706 The authors declare that the data supporting this study's findings are available in
707 the paper and its supplementary information files.
708
709

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- [CommunicationMaterSupplementaryInformation.docx](#)