



# Towards Quantitative Inference of Nanoscale Defects in Irradiated Metals and Alloys

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Quantifying the population of nanoscale defects that are formed in metals and alloys exposed to extreme radiation environments remains a pressing challenge in materials science. These defects both fundamentally alter material properties and seed long-timescale performance degradation, which often limits the lifespan of engineering systems. Unlike ceramic and semiconducting materials, these defects in metals and alloys are not spectroscopically active, forcing characterization to rely on indirect measurements from which the distribution of nanoscale defects may be inferred. In this mini-review, different experimental methodologies which have been employed for defect inference are highlighted to capture the current state of the art. Future directions in this area are proposed, which, by combining data streams from multiple and complementary characterization methods in concert with multi-scale modeling and simulation, will enable the ultimate goal of quantifying the full spectrum of defects in irradiated metals and alloys.

**Keywords:** Defects, irradiated, metals, resistivity, positron annihilation spectroscopy, X-ray scattering, Rutherford backscattering, transient grating spectroscopy

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## INTRODUCTION

Materials in extreme radiation environments—from nuclear energy systems, to particle accelerators, to satellites—experience some of the most demanding sets of conditions for components in-service (Allen et al., 2010; Gilbert et al., 2021). In addition to elevated temperatures, stresses, and corrosive species, materials must withstand fluxes of high energy particles, often over long operational lifetimes. These particles collide with atoms, creating cascades of displacements on very short timescales (Zinkle and Singh, 1993). The formation and evolution of these primary, nanoscale defects eventually leads to microstructural changes across all length scales and results in the degradation of material properties.

Techniques used to investigate structural defects in irradiated materials include, but are not limited to, electron microscopy (Jenkins and Kirk, 2001), optical spectroscopies (Rickert et al., 2022), X-ray and neutron scattering (Albertini and Coppola, 1992; Ehrhart, 1994), ion-beam analysis (Swanson, 1982), field ion microscopy (Seidman, 1978), and positron annihilation spectroscopy (Selim, 2021). While transmission electron microscopy (TEM) has been extensively used to characterize radiation-affected microstructures (Jenkins and Kirk, 2001) due to a practical resolution limit of ~1 nm (Zhou et al., 2007). Simulations show that displacement cascades create a power law-scaled distribution of defect clusters (Yi et al., 2015), implying that the smallest defects are the most prevalent in irradiated metals. A void with a

diameter of 1 nm corresponds to a cluster of ~350 vacancies (Caturla et al., 2000). With TEM unable to reliably resolve clusters of this size and below, electron microscopy drastically underestimates the total defect density in irradiated materials (Meslin et al., 2010; Reza et al., 2020; Ungár et al., 2021a).

In order to understand the mechanisms which govern irradiation-induced changes in properties, it is crucial to characterize the formation and evolution of defects on these smallest scales. In addition to seeding larger scale defect formation, nanoscale defects can have a significant effect on the properties of irradiated materials. Reza et al. show that defects below the resolution limit of TEM play a dominant role in the decrease of thermal diffusivity for self-ion irradiated W (Reza et al., 2020). Li et al. report that the ultrahigh hardening of He-irradiated Nb results from the presence of vacancies (*V*) and He-*V* complexes (Li et al., 2022). Thus, for accurate prediction of irradiated materials' properties at the macroscale, it is critical to characterize defects at the nanoscale.

In non-metals, these defects can often be characterized through optical spectroscopic techniques such as Raman spectroscopy (Shelyug et al., 2018), optical ellipsometry (Khanolkar et al., 2022), photoluminescence (Khanolkar et al., 2022), and others. In Raman-active materials, the vibrational modes of different bonds give unique signatures that can be probed optically and provide insight into defects as small as isolated vacancies and interstitials. Similarly, these point defects in many ceramics are charged, leaving them optically accessible through absorption, excitation, or emission. Due to charge screening from free electrons in metals and alloys, these spectroscopic methods may not be readily applied to the study of defects. Thus, nanoscale defects must be detected through alternative means. Their presence, type, and density can be determined indirectly through their effect on certain material properties and structural features. The purpose of this mini-review is to highlight developments in these inference-based techniques and report progress towards the quantitative characterization of nanoscale defects in irradiated metals and alloys.

## RESISTIVITY MEASUREMENTS

Some of the oldest methods deployed to infer populations of nanoscale defects in metals are those relying on changes in electrical resistivity. Perturbations of the crystalline lattice increase resistivity through enhanced scattering of electrons (Broom, 1954). Net changes in resistivity can be expressed as the sum of contributions from all types of defects under the principle developed by (Matthiessen and Vogt, 1864), which considers contributions from each defect type through their concentration and specific resistivity.

Many studies have used resistivity measurements to infer the Frenkel pair concentration after cryogenic irradiation. These include experiments seeking to determine threshold displacement energies (Lucasson and Walker, 1962), defect production rates, spontaneous relaxation volumes, and the saturation concentration of defects (Nakagawa et al., 1977;

Nakagawa et al., 1979; Nakagawa, 1982). With defect migration deactivated at low temperature, this method has enabled the evaluation of primary damage formation from different irradiation particles in FCC metals (Iwase et al., 1992) and Fe (Chimi et al., 2000), and the validation of displacement cross sections in Cu (Iwamoto et al., 2015).

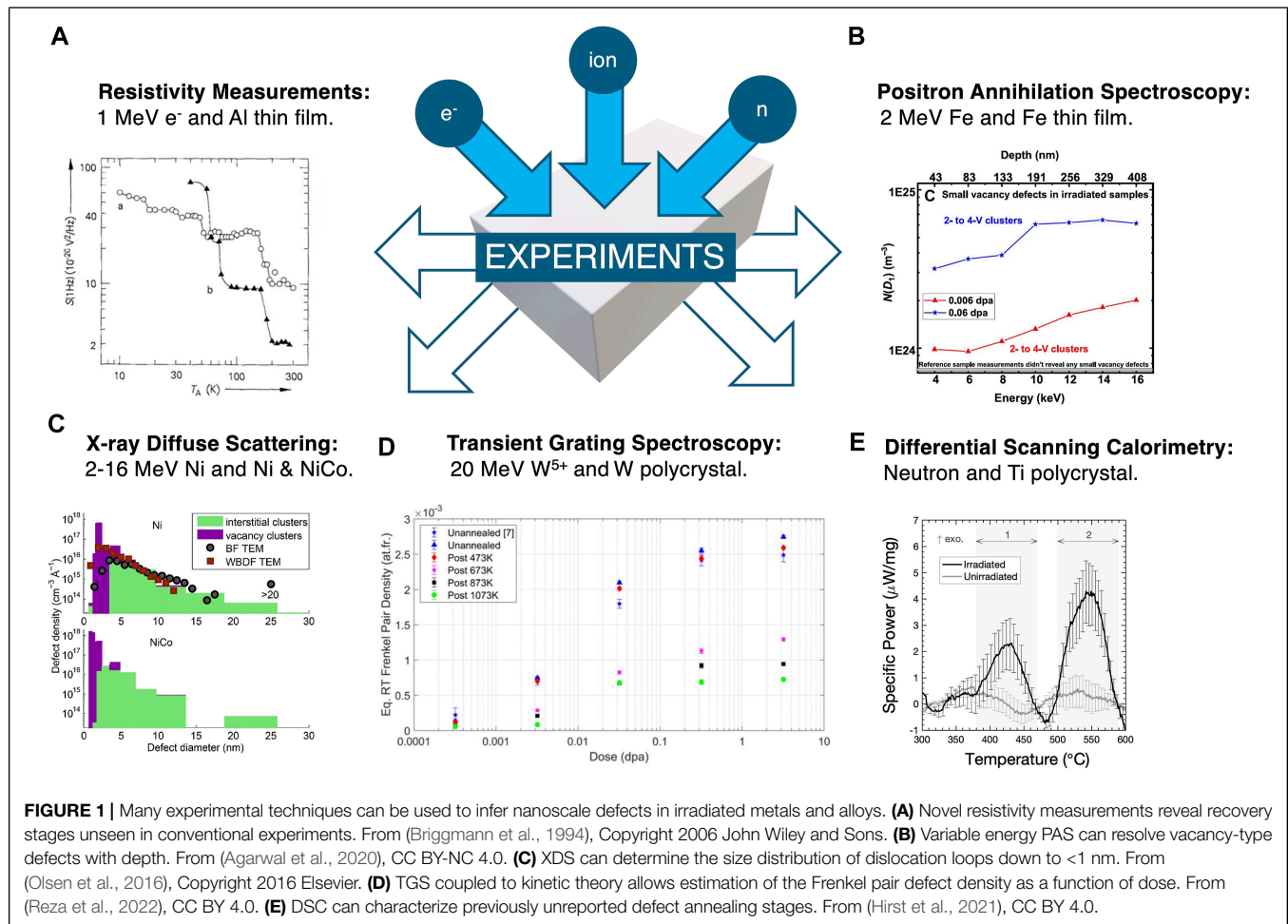
Often these studies also use isochronal annealing to evaluate the recovery of specific defect populations with increasing temperature (Lucasson and Walker, 1962; Nakagawa et al., 1977; Nakagawa, 1982; Iwase et al., 1992; Chimi et al., 2000; Iwamoto et al., 2015; Horak and Blewitt, 1975). These experiments can be coupled to kinetic Monte Carlo (kMC) simulations (Fu et al., 2004; Fluss et al., 2004) of the defect evolution to gain insight into the precise recovery mechanism, shown in **Figure 2A**. Additionally, several studies have correlated resistivity to stored energy measurements in order to validate the change in defect concentration (Kinchin and Thompson, 1958; Isebeck et al., 1966; Delaplace et al., 1968; Nicoud et al., 1968; Losehand et al., 1969). Resistivity measurements have also been used to evaluate the effect of solutes on the recovery of radiation damage, including studies on C-doped Fe (Takaki et al., 1983) and Fe-Cr (Gómez-Ferrer, 2016).

Alternative measurement schemes have also been developed. For example, Briggmann et al. use the  $1/f$  nature of noise from a resistivity measurement to characterize defects through their migration rather than through their annihilation (Briggmann et al., 1994). This method reveals the presence of recovery stages, shown in **Figure 1A**, that do not appear in conventional resistivity measurements, which are attributed to de-trapping of crowdions. Nikolaev has also championed the use of differential resistivity recovery measurements to decouple the effects of changing defect concentration and specific resistivity (Nikolaev, 2007). This has been used to determine the effect of short range order in Fe-based alloys (Nikolaev, 2009), and to deconvolve resistivity contributions from vacancies and interstitials (Nikolaev, 2018).

One major limitation of this family of techniques includes the inability to directly simulate the resistivity of large defects, as density-functional theory (DFT) is computationally-limited to small supercell volumes. Additionally, many instances of deviation from Matthiessen's rule have been reported (Bass, 1972; Fluss et al., 2004). The effect of defect clustering on the specific resistivity has been studied by Zinkle et al. (Zinkle, 1988) who report that the Frenkel pair resistivity for defects in small dislocation loops is similar to that of isolated Frenkel pairs, although this may not hold for larger loop sizes. These factors hinder the accurate determination of defect densities.

## POSITRON ANNIHILATION SPECTROSCOPY

Positrons annihilation spectroscopy (PAS) is able to detect open-volume in a crystalline lattice by virtue of locally reduced electron density, allowing incident positrons to probe vacancy-type defects (Selim, 2021). Similar to resistivity measurements, positron annihilation lifetime spectroscopy (PALS) has been



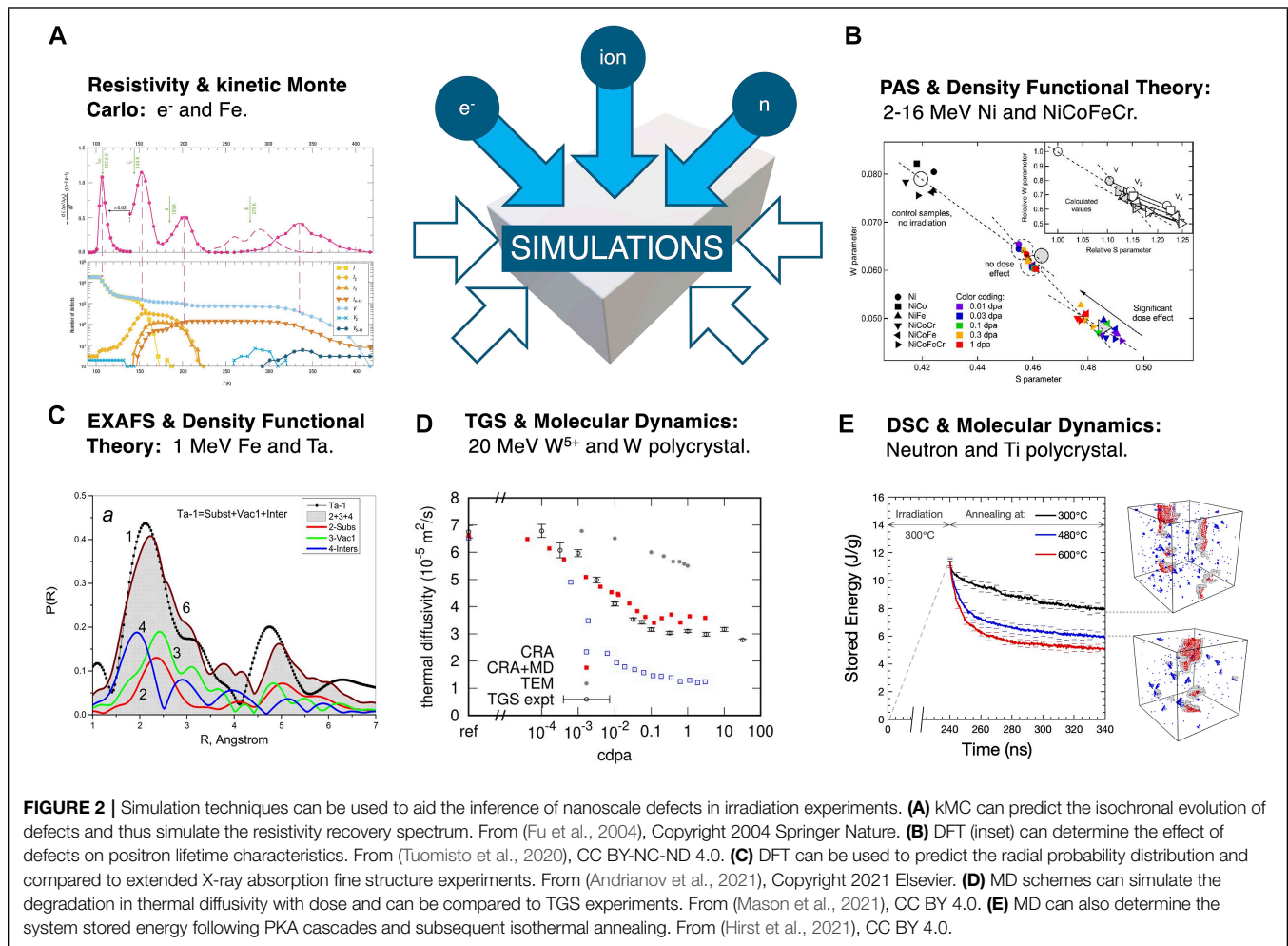
used for many years to investigate the formation and evolution of primary radiation damage in metals after irradiation at cryogenic temperatures and subsequent isochronal annealing (Mantl and Triftshäuser, 1978; Eldrup and Singh, 1997; Eldrup and Singh, 2003). Decomposition of the positron lifetime spectrum into multiple components can be used to characterize the size distribution of vacancy clusters (Eldrup and Singh, 2003; Hu et al., 2016), although there is a limit to the number of components that can be identified.

Additionally, the local chemical environment around vacancy-type defects can be explored through analysis of the momentum of the annihilating electron in Coincidence Doppler Broadening (CDB) experiments (Selim, 2021). This method has enabled the detection of solute-vacancy complexes in electron-irradiated reactor pressure vessel (RPV) model alloys. (Nagai et al., 2003) used CDB experiments to estimate the local concentration of solute around vacancies and attribute the irradiation-induced hardening of RPV steels to the formation of these features.

By varying the energy of the incident positrons, the depth-dependence of defect populations can be resolved in materials with heterogeneous microstructures (Lynn et al., 1986; Siemek et al., 2021). Recently, Agarwal et al. (Agarwal et al., 2020) used this method to probe the depth

dependence of vacancy clusters created following 2 MeV self-ion irradiation of Fe thin-films, shown in **Figure 1B**. Their work reports an increase in the density of small vacancy clusters with depth and an associated decrease in large vacancy clusters. Most strikingly, they report a decrease in the diameter of cavities with increasing dose, which is attributed to a new mechanism of interstitial-induced shrinkage of voids and resultant intra-cascade nucleation of small vacancy clusters.

The ability of PALS to detect individual vacancies allows direct comparisons to be made to primary radiation damage simulations Tuomisto et al. (Tuomisto et al., 2020) studied Ni-ion irradiation damage in NiCoFeCr and its derivative alloys through PALS, molecular dynamics (MD), and density functional theory (DFT) simulations, shown in **Figure 2B**. They compare the modeled fraction of vacancies as a function of dose to their experimental data to rule out potential mechanisms and in doing so reveal the segregation of Ni and Co to vacancies. Soneda et al. (Soneda et al., 2003) use kMC simulations to model defect accumulation in Fe and demonstrate strong agreement between their predicted number densities of vacancy clusters and earlier PAS work. Further information on the use of PAS for irradiation materials can be found in a comprehensive review by Selim (Selim, 2021).



## X-RAY BASED METHODS

### Scattering

X-ray scattering has been extensively used to probe radiation damage in metals due to its sensitivity to defects below the resolution limit of TEM and the ability to directly compare measurements to scattering theory. Many studies have used X-ray diffuse scattering (XDS) to characterize the size distribution of dislocation loops in irradiated Cu (Larson, 1975; Larson and Young, 1987; Ehrhart and Averback, 1989), Ni (Narayan and Larson, 1977; Larson and Young, 1987; Ehrhart and Averback, 1989; Yuya et al., 1999; Olsen et al., 2016), and W (Sun et al., 2018). Most of these studies compare their XDS results to the size distribution of loops determined from TEM measurements. These comparisons explicitly demonstrate a) the presence of a significant fraction of loops with radii below 1 nm, and b) the inability of TEM to characterize this population of defects. An example from (Olsen et al., 2016) is shown in Figure 1C.

More recently, the convolutional multiple whole profile (CMWP) X-ray diffraction Line Profile Analysis (XPLA) method (Ribárik, 2008; Ribárik et al., 2020) has been developed

to characterize dislocation loops in irradiated materials (Seymour et al., 2017; Ungár et al., 2021b). Through calculation of the broadening of X-ray diffraction (XRD) peaks, the type, density, and size distribution of dislocation loops can be determined. Ungár et al. (Ungár et al., 2021a) demonstrated this in proton-irradiated Zircaloy-2 where they combined TEM and synchrotron XRD to calculate the power law size density function of  $\langle a \rangle$ -loops, showing a significant density of loops below TEM resolution. Additionally, they extend their analysis to determine the average loop diameter as a function of loop density. They report, counter intuitively, that initially a low density of large  $\langle a \rangle$ -loops forms due to growth without impingement on other loops. Further work at low doses is needed to validate this claim.

### Absorption

In addition to X-ray scattering, the local environment around atoms can be investigated using extended X-ray absorption fine structure (EXAFS) spectroscopy. EXAFS allows calculation of the radial distribution function which reveals the presence of defects through changes in the local coordination. This enables experiments to be directly compared to simulations, as demonstrated by Andrianov et al. (Andrianov et al., 2021)

in **Figure 2C**. EXAFS has been used to study the impact of self-irradiation in Pu and its alloys as a function of dose (Booth et al., 2007; Booth et al., 2013; Olive et al., 2016; Booth and Olive, 2017). Booth et al. use EXAFS to determine the fraction of a sample that is damaged and compare these measurements to estimates based on point defect production and annealing (Booth et al., 2013). From this comparison, they report that classical defect production models (NRT-dpa and Kinchin-Pease) are insufficient to capture the effect of different bonding on defect evolution in intermetallics. Further validation of this finding with MD simulations — as demonstrated by Okamoto et al. (Okamoto et al., 1991) for N-ion irradiated amorphous  $\text{Pd}_{80}\text{Si}_{20}$ , and Booth and Olive (Booth and Olive, 2017) for pure Pu — would be insightful, but subject to the availability of accurate interatomic potentials.

The ability of EXAFS to probe the local structure also extends to chemical environments. Kuri et al. investigated neutron-irradiated, annealed and re-irradiated, RPV steels to analyze the coordination of solutes Cu, Ni, and Mn (Kuri et al., 2009). They report similar atomic environments for each of the solute atoms, with attenuation in the radial distribution function peaks attributed to local disorder. The nearest neighbor coordination of Fe atoms does not decrease, which suggests the presence of vacancy-solute complexes, as has been demonstrated in PAS (Nagai et al., 2003).

## RUTHERFORD BACKSCATTERING

Rutherford backscattering in the channeling condition (RBS/C) is part of a family of ion beam analysis techniques that have been used to characterize disorder in irradiated materials (Chu et al., 1973; Swanson, 1982; Matzke, 1985). Placing a reference material in a channeling condition under light ion bombardment dramatically reduces the backscattered ion yield. However, by introducing defects or disorder into the lattice via irradiation, that backscattered yield increases. By varying the energy of the incident ions, the depth dependence of radiation damage can be probed. Comparing these data to the predicted damage profile allows for interpretation of the mobility of irradiation-induced defects, as shown in (Lu et al., 2016).

The radiation resistance of Ni-based complex solid solution alloys (CSSAs) has been extensively explored using RBS/C (Zhang et al., 2015; Jin et al., 2016; Lu et al., 2016; Veliş̇a et al., 2017; Fan et al., 2019). Through comparison of the backscattering yield of different alloys irradiated to the same dose, the effect of chemical complexity on recombination can be deduced. The morphology of larger defect clusters is found to evolve as a function of dose, with dislocation loops forming at higher dose levels, which reduce the irradiation-induced strain in the material (Jin et al., 2016; Veliş̇a et al., 2017).

Most studies use measures of the relative disorder between pristine and as-irradiated specimens to investigate the accumulated defects. However, further insight into nanoscale defect populations can be gained from RBS/C spectra simulated from atomistic configurations (Zhang et al., 2016; Zhang et al., 2017; Levo et al., 2021). Zhang et al.'s simulations

of ion-irradiated Ni indicate that the contribution to the RBS/C signal from extended defects is stronger than the contribution from Frenkel pairs. While this appears to conflict with previous studies which report a decrease in irradiation-induced strain and thus decrease in backscattering signal with defect agglomeration, this work demonstrates the insight that can be gained from a combination of RBS/C experiments and simulations.

## TRANSIENT GRATING SPECTROSCOPY

Recently, local thermophysical properties as measured using transient grating spectroscopy (TGS) have been applied to the measurement of radiation effects on metals and alloys (Short et al., 2015; Dennett et al., 2016; Hofmann et al., 2019). In these experiments, a micron-scale 1D periodic laser excitation causes local heating and thermoelastic generation of surface-confined acoustic waves (Käding et al., 1995; Johnson et al., 2012; Hofmann et al., 2019). By optically measuring the dynamics of these excitations, elastic and thermal properties can be extracted simultaneously from a single non-destructive measurement (Dennett and Short, 2018). The measurement sampling depth is on the order of single microns, a length scale particularly relevant for the study of metals with defects induced through ion beam irradiation (Hofmann et al., 2015a; Hofmann et al., 2015b; Dennett et al., 2018; AlMousa et al., 2021). Capabilities for *in situ* TGS measurements during high temperature ion beam exposure have also been developed (Dennett et al., 2019).

TGS has been used for nanoscale defect inference in several studies. Ferry et al. used changes of thermal diffusivity in Si-ion irradiated Nb to infer the agglomeration of point defects into clusters, reducing total thermal carrier scattering (Ferry et al., 2019). The most direct demonstration of nanoscale defect inference through TGS has come from Reza and coworkers (Reza et al., 2020; Reza et al., 2022). Initially, measurements of thermal diffusivity in self-ion irradiated W specimens were used in concert with a point defect-electron scattering model to infer the population of Frenkel defects retained after room temperature irradiation to dose levels spanning five orders of magnitude (Reza et al., 2020). Comparing with TEM-measured defect populations, a total defect density approximately an order of magnitude higher across all conditions was inferred. By using MD defect generation simulations to predict the nanoscale defect populations missed by TEM, the TGS-inferred and TEM/MD combination defect densities become comparable. In follow-on work, Reza et al. use *in situ* TGS during thermal annealing of previously-irradiated samples to infer point defect densities and explore their recombination kinetics (Reza et al., 2022), shown in **Figure 1D**. They then compared their work to MD estimations of the thermal diffusivity degradation with increasing dose, shown in **Figure 2D**.

Using local elastic properties measured with *in situ* TGS during high temperature ion irradiation, Dennett et al. studied the accumulation of point defects, vacancy clusters, and eventually void swelling in a series of Ni-based CSSAs up to and including 5-component, single phase Cantor alloy

(Dennett et al., 2021). Alloy chemistries known to retain a higher density of nanoscale vacancy clusters, such as ternary NiCoCr, showed a much stronger elastic signature of vacancy-type defect density. Although TGS returns both elastic and thermal properties simultaneously, no work has yet demonstrated an inference method based on the combination of these properties together.

## CALORIMETRY

The characteristic energies of formation and migration can be used to infer the density of irradiation-induced defects through calorimetric experiments. Annealing irradiated materials leads to multiple stages of recovery (Schilling, 1978) where the recovery onset temperature can be used to determine the migration energy, therefore defect type, and the enthalpy change divided by the formation energy, therefore defect density. Due to their high stored energy density ( $\sim 10^3$  J/g) (Snead et al., 2019) most prior literature focuses on the recovery of irradiated ceramics, including studies on graphite (Iwata, 1985),  $\text{UO}_2$  (Staicu et al., 2010), SiC (Snead et al., 2019), and NaCl (Vainshtein and Hartog, 2000). However, this strategy is also applicable to metals.

After Eugene Wigner first postulated that energy could be stored in metals as irradiation-induced defects (Wigner, 1942), many works investigated defect recovery at cryogenic temperatures. These include studies on Cu (Losehand et al., 1969; Blewitt et al., 1959; Richard et al., 1990), Al (Isebeck et al., 1966), Mg (Delaplace et al., 1968), and Be (Nicoud et al., 1968). Often the focus of these works was the fundamental science behind primary radiation damage production, rather than investigating defect evolution at engineering-relevant temperatures. Research has been conducted following ambient temperature irradiation, including studies on Mo (Kinchin and Thompson, 1958; Pedchenko and Karasev, 1971; Lambri et al., 2009), Cu (Pedchenko and Karasev, 1971; Blewitt et al., 1961), Ni and Fe (Toktogulova et al., 2010), and Pu (Ennaceur and Migliori, 2018). But little work has been conducted following irradiation at elevated temperatures, with Lee et al.'s analysis of Zr-U (Lee et al., 2007) and Hirst and colleagues' recent study of Ti (Hirst et al., 2021), shown in **Figure 1E**, the only research to date. In addition, very few prior studies have leveraged the information contained within the fine structure of annealing spectra, with the exception of (Richard et al., 1990). The authors of this study derived a kinetic model which was fit to the

experimental recovery of neutron-irradiated Cu. This model yielded information on the activation energies of multiple substages and was used to (in)validate the proposed recovery mechanism for reactor-irradiated metals.

Calorimetric measurements have the key advantage of being directly comparable to simulations of radiation damage. However, until recently, this comparison had only been conducted for ceramics (Béland et al., 2013). Hirst et al. used MD simulations to investigate the evolution of defects below the resolution limit of TEM in neutron-irradiated Ti (Hirst et al., 2021). Their isothermal annealing simulations, shown in **Figure 2E**, reveal a new mechanism for radiation damage recovery, with dislocation loops sweeping up point defects as they glide. This process was correlated to differential scanning calorimetry (DSC) measurements of the stored energy release between 300 and 480°C, where TEM micrographs show little change in the microstructure. This work reports that TEM is unable to account for 80% of the stored energy release and demonstrates the use of DSC as a tool to characterize defects that may be hidden to microscopy techniques.

## FUTURE DIRECTIONS

**Table 1** compares some characteristic features of each of the techniques highlighted above. While great insight into the populations of nanoscale defects can be gained from these methods, their accuracy depends on the quality of data interpretation. Without the ability to directly image defects, inference-based techniques rely on theoretical models to deduce the type, size distribution, and number density of defects in materials.

One strategy that can be used to strengthen the inference model is to combine data streams from multiple methodologies. Disparate techniques often have unique sensitivities to different defect features, offering some degree of measurement orthogonality. Correlative experiments thus allow for a more comprehensive characterization of defect populations. This can be done *ex situ*, as in Meslin et al.'s study of neutron-irradiated ferritic alloys (Meslin et al., 2010), or *in situ*, demonstrated by Jackson's simultaneous measurement of resistivity recovery and stored energy release in deuteron-irradiated metals (Jackson, 1980).

Many inference-based techniques are non-destructive, which allows for both concurrent multi-modal characterization and also the ability to observe nanoscale defect generation and evolution

**TABLE 1** | Comparison of some salient features of the reviewed defect inference techniques. All methods listed can be applied to both pure metals as well as alloys and for irradiation with both neutrons and ions. Note: classifications should be understood as generalizations and abilities thus far demonstrated, not as absolute limitations.

	Resistivity	PAS	X-ray	RBS/C	TGS	Calorimetry
Max. probing range	cm	mm	cm	10 $\mu\text{m}$	10 $\mu\text{m}$	cm
Polycrystalline samples?	✓	✓	✓ (XLPAs)	✗	✓	✓
Point/Extended Defects	Point	Point	Both	Both	Both	Both
Vacancy/Interstitial	Both	Vac	Both	Both	Both	Both
Chemical sensitivity?	✓	✓ (CDB)	✓ (EXAFS)	✗	✓	✓
Destructive?	✗	✗	✗	✓	✗	✓

in real-time. Combining inference-based techniques and ion-accelerators allows for significant insight, but requires detailed consideration of the experimental geometries, as highlighted by Dennett et al. (Dennett et al., 2019). The ability to investigate the dynamics of defect accumulation and recovery *in situ* can uncover fundamental mechanisms and also accelerate nuclear materials development. It is under this promise that recent efforts have been directed in this area (Selim, 2021) and continued expansion of such capabilities is a necessity.

Combining experimental methods with simulated data, as shown in **Figure 2**, can also greatly benefit the interpretation of inference-based methods. This can be done directly, as in the comparison of simulated and measured atomic radial distribution functions (Andrianov et al., 2021), or indirectly, through the measurement and estimation of thermal diffusivity degradation using TGS and MD (Reza et al., 2020; Mason et al., 2021). The limitations of this strategy remain the considerable range of time and length scales that radiation damage encompasses. Atomistic simulations are typically restricted to timescales below microsecond and coarser methods rely on accurate parameterization. Recent advances in kMC methods (Béland et al., 2015) have demonstrated the ability to couple displacement cascades to experimental timescales and warrant further study.

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- In summary, the development of inference-based techniques has significantly advanced the ability to characterize the entire spectrum of defects formed in metals and alloys under irradiation. However, truly quantitative measurement of defects at the smallest scales remains a grand challenge. By designing future experimentation and simulation to function in concert, rapid progress towards this goal may be achieved.

## AUTHOR CONTRIBUTIONS

CAD conceived the manuscript scope. CAH wrote the first draft of the manuscript. CAH and CAD jointly revised and approved the manuscript for publication.

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