

Does uncertainty in microscopy techniques affect significantly the embrittlement trend curve of reactor pressure vessels?

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ABSTRACT A semi-quantitative study was conducted to determine how systematic errors in microscopic analysis in structural materials under neutron irradiation affect their evaluation of ageing degradation.

INTRODUCTION

The timing and intervals of reactor pressure vessel (RPV) surveillance tests are determined by embrittlement trend curve (ETC), which predicts neutron irradiation embrittlement of RPV materials. ETC usually takes into account material change mechanisms corroborated by microscopic observations. The latest Japanese ETC (Hashimoto, *et.al.*, 2021) features well consistency with the nanostructural changes in the surveillance materials, which are obtained exhaustively using 3-dimensional atom probe tomography (APT). However, cutting-edge microscopy techniques are subject to systematic measurement errors called "artifacts" inherent to the equipment and analysis means. This study verifies whether the artifacts have a sufficiently small impact on the ETC from a materials science perspective.

METHODS

APT targets each atom in a tiny sample and obtains its three-dimensional coordinates and mass number. There is a trade-off between APT's yield (tens of percent of total atoms) and elemental (and positional) resolution. APT data from following three researches:

- a) actual surveillance materials (Murakami, 2020);
- b) ion-irradiated RPV model with dislocation segregation; and
- c) numerical simulation assuming the measurement errors (Pan, 2023);

are analyzed.

Two algorithms, maximum separation method (MSM) and iso-position method (IPM), were used to extract characteristic regions from the atoms distribution. A sensitivity analysis of the search parameters, effects of atomic yield, and elemental resolution were evaluated. APT data are characterized by the number density, volume fraction, composition, and diameter of solute atom aggregates, which is a major cause of the RPV embrittlement.

The correlation between embrittlement and APT results was then discussed.

RESULTS

As shown in Figure 1, the impact of APT resolution on the analysis results was quantitatively evaluated using MSM. The data used were hypothetical, with atoms randomly positioned at concentrations consistent with APT observation conditions, simulating the composition of the spherical nanostructures observed in the RPV, which were then assigned to iron and solute elements. APT is an excellent technique that allows elemental analysis at the nanoscale, but is known to systematically misidentify elements. Therefore, we assumed a 5% probability of misrecognition of solute element and iron. The solute clustered regions were then identified by MSM. In other words, an error of about 5% is expected in the volume considered as a cluster. However, as shown in Figure 1 (b), the volume of the clusters were overestimated by the given noise, and this tendency was greater for smaller clusters. This is an artefact caused by the algorithm of MSM.

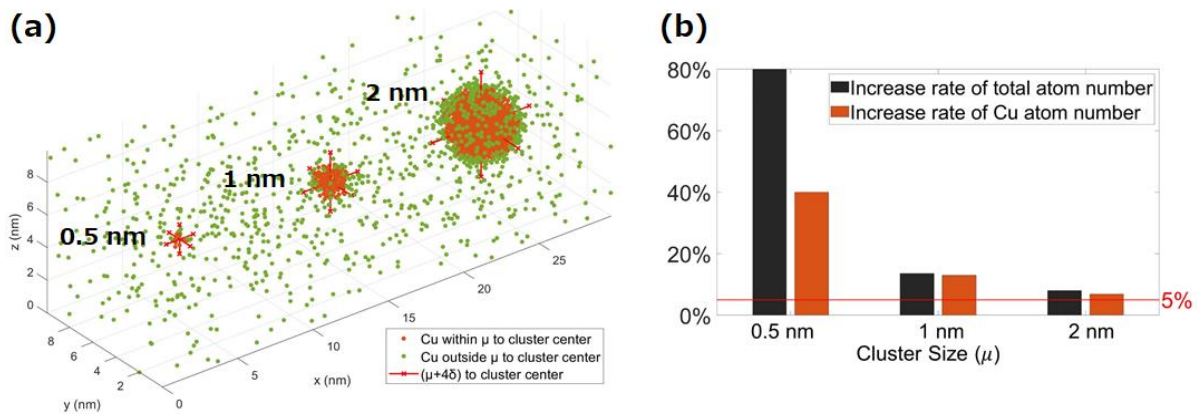


Figure 1. Aggregation of solute atoms searched by MSM from numerically simulated APT data (Pan, 2023)

Trends on sensitivity analysis also vary by algorithm. The MSM, which is used in the development of the Japanese ETC, uses the distance of cluster search as a major parameter to determine the volume to be identified as clusters. IPM, on the other hand, specifies a threshold concentration (C_{min}) as a parameter in addition to the distance of cluster search. As shown in Figure 2, the number density increases with the search distance in MSM, while in IPM the search distance is robust to the number density.

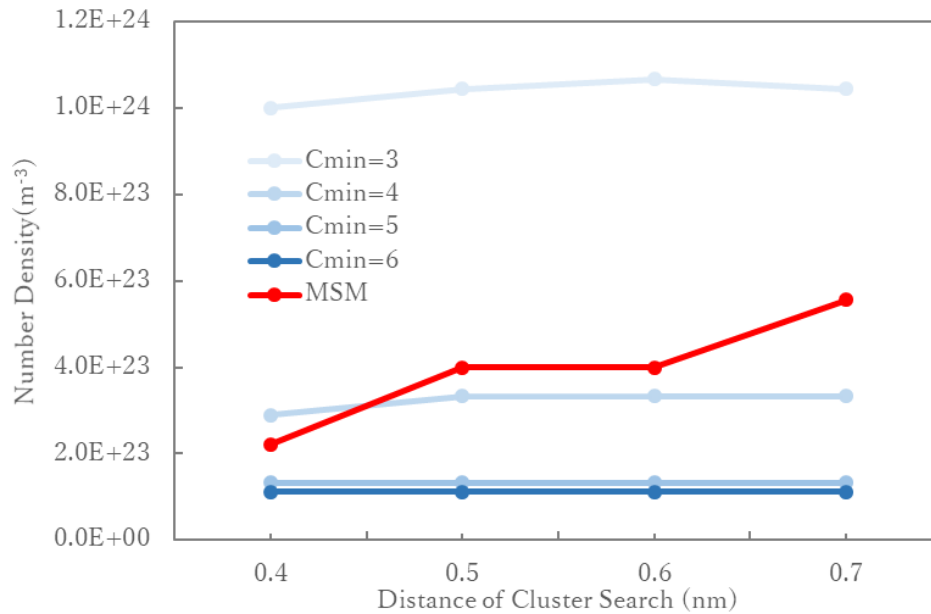


Figure 2. An Example of Sensitivity Analysis of Search Parameters of Clusters in Different Algorithms (Miyake, 2023)

CONCLUSION

Although systematic artifacts were observed in the APT results, their impact on the ETC was found to be relatively small. The presentation will also provide a semi-quantitative explanation of the impact on the irradiation hardening model.

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