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Elemental distribution in a decommissioned high Ni and Mn reactor pressure vessel weld metal from a boiling water reactor

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ABSTRACT

In this paper, weld metal from unique material of a decommissioned boiling water reactor pressure vessel is investigated. The reactor was in operation for 23 effective full power years. The elemental distribution of Ni, Mn, Si and Cu in the material is analysed using atom probe tomography. There are no well-defined clusters of these elements in the weld metal. However, some clustering tendencies of Ni was found, and these are interpreted as a high number density of small features. Cu atoms were found to statistically be closer to Ni atoms than in a fully random solid solution. The impact of the non-random elemental distribution on mechanical properties is judged to be limited.

1. Introduction

The integrity of the reactor pressure vessel (RPV) is of great importance for the safety of a nuclear power plant [1–3]. During operation, neutrons from the reactor core interact with the material and affect the mechanical properties. During the lifetime of the power plant, the RPV is embrittled and the ductile-to-brittle-transition-temperature (DBTT) is increased [3,4]. This change in properties is due to several phenomena taking place in the material. The neutrons interact with the steel, and the result of this is formation of matrix defects (such as clusters of vacancies and interstitials). Furthermore, P segregates to grain boundaries, and nanometre sized clusters containing Ni, Mn, Si, and Cu are formed in the material. This has been studied for a wide range of RPV materials and conditions [5–15].

In order to experimentally study the RPV, surveillance material is often used. This material has been placed inside the reactor during operation, and can thus be removed at different times, to study the effect of irradiation at certain neutron fluences. The surveillance samples are placed closer to the core and are thus exposed to a higher neutron fluence and flux than the RPV itself. This makes it possible to predict what occurs in the actual RPV in advance, although there might be some effects of the higher flux [16–21]. There are few studies of the RPV other than surveillance material, as the RPV is not possible to replace [1], and thus a decommissioned RPV is a unique asset [22,23]. Atom probe tomography (APT) enables to study the Ni, Mn, Si, and Cu clustering, as the chemical composition can be studied with near-atomic resolution using this technique [24].

Earlier studies of Uddcomb manufactured Swedish high Ni and Mn, low Cu surveillance weld metal has shown that the composition results in excellent properties at the beginning of life, but a significantly higher than expected embrittlement compared to many other active nuclear reactors in pressurized water reactors (PWRs) during neutron irradiation [25,26]. This is attributed to the high Ni and Mn content of the material. APT analysis reveals nanometre clusters that are mainly rich in Ni, Mn and Si, and only contain small amounts of Cu [27–29]. Positron annihilation spectroscopy (PAS) of surveillance material during annealing show dissolution of vacancy clusters at 650 K and dissolution of solute–vacancy clusters at 750 K [30]. Furthermore, the effect of neutron flux relevant for PWRs has been studied for these materials by comparing surveillance material with high flux materials research reactor irradiated material. It was found that although a higher flux resulted in smaller clusters with a higher number density and more matrix damage, the resulting mechanical properties were similar for the same neutron fluence [28,31,32]. The effects of thermal ageing at higher temperatures have been studied for the same materials, by studying a pressurizer that had been in operation for 28 years at 345 °C. This

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material was embrittled and also, it contained precipitates that contained more Cu than the precipitates in irradiated weld metal [33,34]. Furthermore, studies of the RPV head of Barsebäck Unit 2 show that for that component, that was thermally aged at 288 °C, there is no significant effect on fracture toughness from the ageing [35,36].

In this paper, RPV weld metal from the beltline of a decommissioned RPV from a power plant (Barsebäck unit 2) is analysed using APT. This RPV has high Ni and Mn, and low Cu, like the rest of the Swedish Uddcomb RPVs [37]. Barsebäck Unit 2 was a boiling water reactor (BWR), and thus the RPV has a larger radius than the RPV of a PWR. The result of this is a relatively low neutron fluence and a low neutron flux. The reactor was in operation between 1977 and 2005. The mechanical properties were studied by Lindqvist et al., showing that the Barsebäck Unit 2 weld metal follow ASTM E900 embrittlement trend curve predictions, i. e. the low neutron fluence resulted in low embrittlent [38]. The objective of the project presented here was to study the elemental distribution within the weld metal on the nanometre scale, to investigate whether there is any ongoing clustering of elements as a result of the years of operation with very low neutron flux exposure in a BWR. The investigation is relevant to the operating fleet as there are a number of RPV beltline welds with similar composition still in operation.

2. Material and methods

The material investigated origins from the beltline of the decommissioned Barsebäck Unit 2 BWR. The plant was in operation for 23 effective full power years at a temperature of 270–280 °C. In this time, the material was exposed to a neutron fluence of 7.94 10^{21}n/m^2 at a flux of 1.1 10^{13}n/m^{2}s (E greater than 1 MeV). The RPV was manufactured by Uddcomb and post weld heat treatment (PWHT) at 620 ± 15 °C was performed after welding. As reference material, unirradiated surveillance material was used.

Due to the material being slightly active, specimen preparation was done by focused ion beam/scanning electron microscope (FIB/SEM) lift-out rather than by electropolishing, to reduce the manual handling of the material. The FIB/SEM used was an FEI Versa 3D. Reference material was electropolished by a standard two-step method.

APT analysis was mainly performed in an IMAGO LEAP 3000X HR with a detection efficiency of 37%. Voltage pulsing was used, with a temperature of 70 K, frequency of 200 kHz, and a voltage pulse fraction of 20%. For the data presented in supplementary material, an additional instrument (LEAP 5000 XS) was used, see supplementary material section 1.2. Reconstruction and analysis were done in IVAS 3.6 (Cameca). For the composition, the overlapping peaks were carefully deconvoluted. For the radial distribution functions (RDFs), the peak at 29 Da was assigned as Ni, although it is overlapping with a minor isotope of Fe. Both Ni and Fe ions are evaporated mostly as 2 + ions, as can be seen in the mass spectrum in supplementary material. Thus, 80 % of the 29 peak is Ni, taking into account the composition of the material and the natural abundances of Ni and Fe isotopes.

The RDF was used as a tool to evaluate the distribution of elements within the reconstructions. The RDF is a versatile and parameter free method to understand if elements are randomly distributed or not, and can be used for instance for clustering/precipitation and evaluation of spinodal decomposition [39–42]. The RDF is the average of the normalised composition of a specific element, around another specific element. For instance, the normalised Ni-Cu RDF is the average of radial Cu concentration profiles measured from each Ni atom. Unity indicates an average composition, that means random distribution. Larger values than unity for small distances indicate some kind of clustering or precipitation.

In the supplementary material, there is an extensive discussion on analysis conditions, with an example of laser pulsed analysis, and of a straight flight path instrument.

3. Results

The composition from APT measurements can be seen in Table 1. In total, five analyses were performed, two of the reference material and three of the actual Barsebäck RPV material. As expected, there was no significant compositional difference between the reference and the irradiated material, and thus they are presented together. Due to the inhomogeneous nature of the weld metal, the composition varies between the analyses. This can be seen in the standard deviations given in the table. Some of the values are smaller than the bulk value, as larger particles such as carbides and MnS, and segregation of some elements (for instance Mn, Mo, C, and P) to grain boundaries and dislocations is not taken into account in the values from APT, where only metal matrix is considered. Furthermore, small amounts of some elements (Sn, S) are hard to determine by APT due to overlaps with other elements in the mass spectrum.

The APT data showed no pronounced clustering in the irradiated material. Examples of the Ni, Mn, Si and Cu distributions for both reference and the decommissioned RPV material can be seen in the 10 nm thick slices in Fig. 1. Using isoconcentration surfaces, no clustering could be discerned. Frequency distribution analysis did not show any difference from random for the aged material considering Ni, Mn, Si and Cu atoms, but this is not a well-suited method for the potentially very small clusters due to the division into relatively large blocks. The maximum separation method (MSM) that is commonly used for cluster identification in APT data did not give any indication of significant clustering. The RDF is based on concentration variations, contrary to MSM that is density based. This makes RDF less influenced by density variations, which can induce noise into the MSM. Thus, RDFs were used to characterise the atomic distribution of elements.

The Ni-Ni RDFs for the three irradiated and the two reference material analyses can be seen in Fig. 2 a). The Ni-Ni RDF values for small distances (below about 1 nm) are higher for the aged material than for the reference material. In b), c) and d), the Ni-Cu, Ni-Mn, and Ni-Si RDFs are shown (here Ni is the centre atom, and the normalized concentrations of the other elements are shown relative to the distance to the Ni atoms). The Ni-Cu RDFs indicate that there is a tendency for Cu atoms to be closer to Ni atoms than random for the aged material. In the reference material, there is no such trend. For Mn, the same trend can be seen in two of the analyses of aged materials, whereas the third analysis shows a similar tendency as the reference material. The Ni-Si RDF is higher than reference for one of the aged materials, making the interpretation of the result more challenging. The scatter in the RDF data for small distances is larger for Ni-Cu and Ni-Si than for Ni-Ni and Ni-Mn, as the Cu and Si contents are considerably lower than the Ni and Mn contents, resulting in fewer counts per data point.

Table 1
Compositions, as measured by APT, and average values (measured by optical emission spectroscopy). The error given is the standard deviation between the five analyses (both reference and RPV weld metal) used for the average value.

Element	APT, measured matrix (at.%)	Average bulk value (at.%)
Fe	Balance	Balance
Ni	1.60 ± 0.15	1.39
Mn	1.25 ± 0.06	1.55
Si	0.31 ± 0.09	0.44
Mo	0.18 ± 0.03	0.26
C	0.09 ± 0.08	0.39
Cu	0.05 ± 0.01	0.06
Cr	0.07 ± 0.05	0.14
Co	0.02 ± 0.01	0.01
N	0.04 ± 0.02	–
P	0.02 ± 0.01	0.02
V	0.003 ± 0.001	–
Al	–	0.01
Sn	–	0.003
S	–	0.007

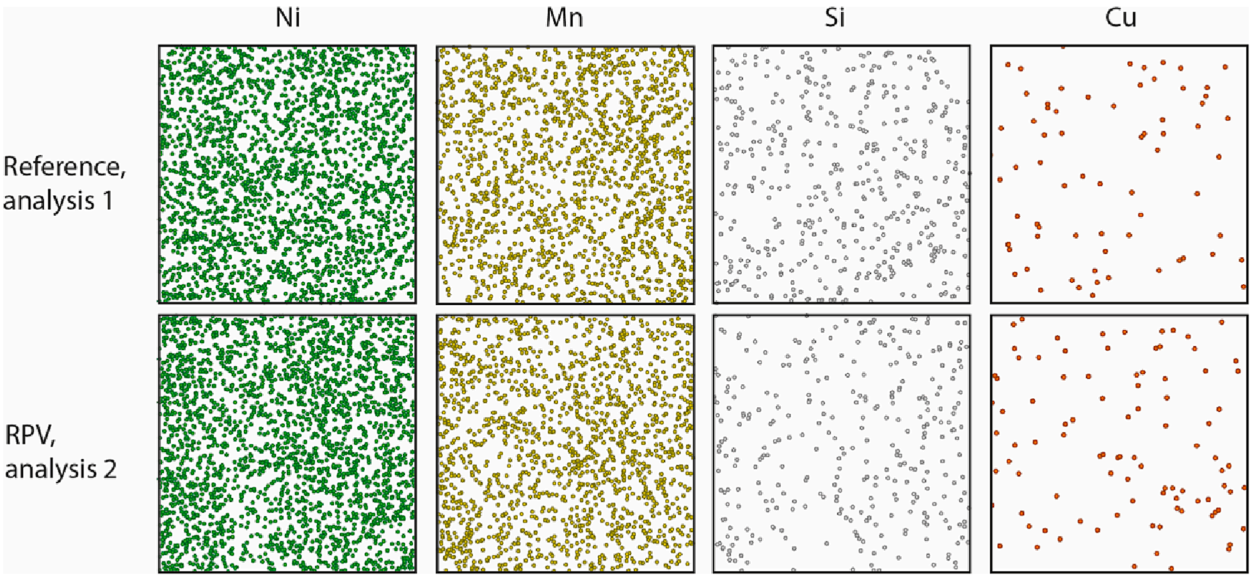

Fig. 1. 20x20x10 nm^3 boxes cut out from APT reconstructions of one reference and one decommissioned RPV weld metal. Ni, Mn, Si, and Cu atoms are shown separately. All detected atoms of these elements are shown.

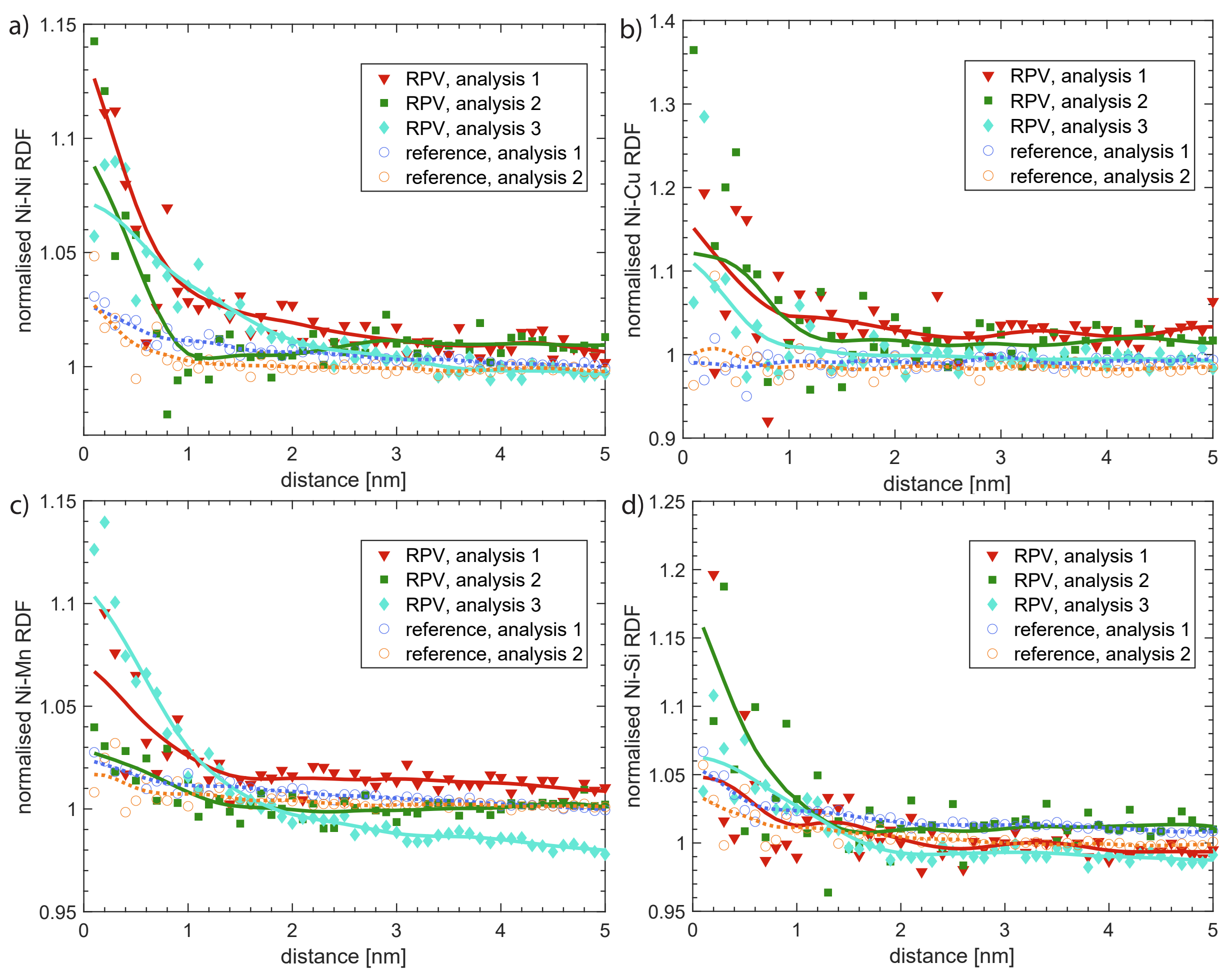

Fig. 2. Normalised a) Ni-Ni RDFs, b) Ni-Cu RDFs, c) Ni-Mn, and d) Ni-Si for voltage pulsed APT reconstructions. The lines are smoothed trend lines to help guide the eye. Solid lines and filled symbols are representing the decommissioned RPV data, and dotted lines and open symbols the reference data. Note the different ranges of the y-axes in the graphs.

4. Discussion

4.1. General remarks

In this paper, it was found that the RPV weld metal of a decommissioned BWR did not show any significant clustering. However, there are some indications of irradiation damage in the Ni distribution, as Ni atoms appear to be clustered when comparing the RDF of the aged material with the reference. This tendency is small and could not be easily observed directly in the data or by isoconcentration surfaces. There is also a tendency of Cu atoms to be closer to Ni atoms than for a random distribution.

The results can be compared with other BWRs. Murakami analysed a surveillance material irradiated to a fluence of 9.4 10^{21}n/m^2 during 22 effective full power years [43], very close to the fluence of the samples of the present study. The material, containing 0.13 at.% Cu in the bulk measured by APT (nominal value 0.24 wt%), contained clusters with a number density of 3.1 10^{23} /m^3. The clusters contained Ni, Mn, Si and Cu. In the heat affected zone from the cladding in an RPV of a decommissioned BWR power plant, Yuya et al. found Ni, Mn, Si and Cu enriched clusters [44]. The A533B steel with 0.09 wt% Cu was exposed to 1.5 10^{22}n/m^2, and the resulting cluster number density was 2 10^{22} /m^3. There are also BWR RPVs in the Japanese database of APT data used for embrittlement trend curves containing clusters [45]. Using atom probe field ion microscopy (APFIM), Burke et al. analysed type A533B plate and weld, irradiated to 2 10^{21}n/m^2, with Cu contents of 0.13 and 0.27 at.% [46]. These did not show any signs of clustering of the same elements (although the analysis volume is small due to the older instrumentation). In another material, exposed to 2.7 10^{22}n/m^2 and containing 0.14 at.% Cu, the same authors did observe one Cu-rich feature.

The effect of composition is complex, and all of the above-mentioned materials are lower in Ni (and most of them in Mn) than the material investigated in this paper. For RPV material irradiated to higher fluences, these compositional effects are well-known. For instance, the low Cu content of similar materials often results in clusters with low or almost no Cu content, whereas a higher Cu content gives Cu-rich clusters [47,48]. It is not unlikely to hypothesise that potential clusters in this low Cu, high Ni and Mn material would contain mostly Ni, Mn, and Si, as the Uddcomb manufactured PWR RPV weld metals do [27–29,31]. Clusters found in Swedish RPV weld metal neutron irradiated to PWR relevant fluences contain mostly Ni (around 50 %), and significant amounts of Mn (around 40 %). The Si and Cu contents found was significantly lower, less than 10% Si and 2 % Cu [28,31]. Thus, the expectation, if the clustering tendencies of the Barsebäck material is in line with the clusters in the PWR irradiated material, is that the Ni-Ni RDF would be the highest, as is the case here. The Ni-Cu RDF also reveals a Cu contribution to the clusters. Mn and Si trends in the data are less distinct but might imply a tendency for Mn and Si atoms to be close to Ni atoms as well.

The neutron fluence is, however, not the only parameter that is different from most analyses that are performed on PWR RPVs. Also, the neutron flux is significantly lower. For PWR relevant fluences and fluxes and low lead factors, the embrittlement is similar [16,17,20,49]. The cluster characteristics might vary slightly. For the Swedish PWRs, it was found that a higher flux (lead factor of up to 75) resulted in smaller clusters with a higher number density than the surveillance material (lead factor of around 3) [28]. For very high neutron fluxes, the embrittlement might be lower for the same fluence at lower flux, as is the case for a BWR surveillance material with a lead factor of more than 150 [37]. The low fluence in combination with the low flux in BWR compared to PWR has, in the Barsebäck RPV weld metal, not resulted in any significant clustering of Ni, Mn, Si and Cu. The tendencies are interpreted as many small rather than few larger clusters (that would be visible in the APT data sets).

Small angle neutron scattering (SANS) studies of the decommissioned nuclear power plant in Greifswald reveal that the extent of clustering in surveillance and actual RPV is similar for this VVER440 (230) type RPV. However, there is a difference in the ratio of magnetic and nuclear scattering of the clusters, implying a difference in composition between the irradiation conditions [23].

The temperature in a BWR is slightly lower than that in a PWR. A lower temperature generally results in a larger effect from radiation as the damage is not annealed out as quickly. On the other hand, the thermal diffusion is lower at lower temperatures. The temperature is estimated to be between 270 °C and 280 °C between the RPV and the core barrel in the beltline region of the BWR studied here. The difference to the Ringhals surveillance material (PWR) that was irradiated at 290 °C is considered a small factor in the context.

The temperature itself is also considered too low for significant diffusion to take place. As a comparison, the PWR RPV head of Ringhals Unit 3, that had been thermally aged at 310–315 °C for 176,000 h (between 1981 and 2005), contains occasional clusters containing Ni, Mn and Cu, but with a very low number density ($<10^{22}$ /m^3), and mainly found on dislocations and boundaries [33]. Thermal diffusion at the relevant temperatures is estimated to be low. The diffusion of Ni in α-Fe during 23 years at 270–280 °C is 0.5—–0.8 nm, but at 310–315 °C 3.0—–3.8 nm when estimated with diffusion constants from [50]. Thus, the effect of thermal ageing only, is estimated to be negligible on the Ni distribution.

The neutron spectrum is different between PWRs and BWRs. The effects of this could be a factor but is not further discussed here.

4.2. Interpretation of the RDF data

In the weld metal from the decommissioned BWR RPV, the Ni-Ni RDF shows a tendency for clustering. This tendency is larger than in the reference material, that has not been irradiated. However, there is a small tendency in the reference material as well, as there is a very small increase for small distances in the RDF, see Fig. 2. This is likely an artefact in the APT data that stems from the field evaporation process. During field evaporation, the crystallography of the material makes the Ni atoms evaporate slightly unevenly across the surface, resulting in some diffuse features throughout the reconstruction. These are easily distinguishable as they follow the Z direction of the analysis. When the datasets are randomised, this effect disappears, and the RDFs are unity all the way to zero distance, with the exception for noise due to the limited volume.

The increase in the Ni-Ni RDF data could be a sign of early clustering. By the use of the parameter free method by Zhao et al, the RDF could be translated into cluster characteristics [39]. Assumptions made are that all clusters have the same size, and that there is no significant depletion around the clusters. As input for this method, the RDF as well as the Ni content and Ni matrix content are needed. To get a matrix Ni content is not straightforward in this case, as the matrix could not be easily discerned from the clusters. Thus, a range of Ni matrix contents is used, to obtain a range of reasonable clusters characteristics. The resulting number density, cluster Ni content, and number of Ni atoms detected per cluster can be seen in Fig. 3. The number of Ni atoms is calculated from the size and cluster Ni content, assuming an even density throughout the reconstruction. It is probable that the Ni matrix content is close to the total Ni content (that is 2.17 at.% for this specific analysis). The 29 peak is ranged as Ni although there is also a contribution from Fe. By deconvoluting the peak, the Ni content becomes 1.74 at.%, which is a better measurement of the real Ni content, but for this RDF method the matrix concentration corresponding to the ranging used for the RDF has to be used. This would give number densities in the order of 10^{23} – 10^{24} /m^3. This is in accordance with the number densities of similar RPV weld metal neutron irradiated to higher fluences at higher neutron flux [28,31]. If there is clustering in the data, the clusters would be too small to easily be seen in the APT data, thus the number of Ni atoms per cluster should be reasonably small. As an example, a matrix content of 2.14 at.%

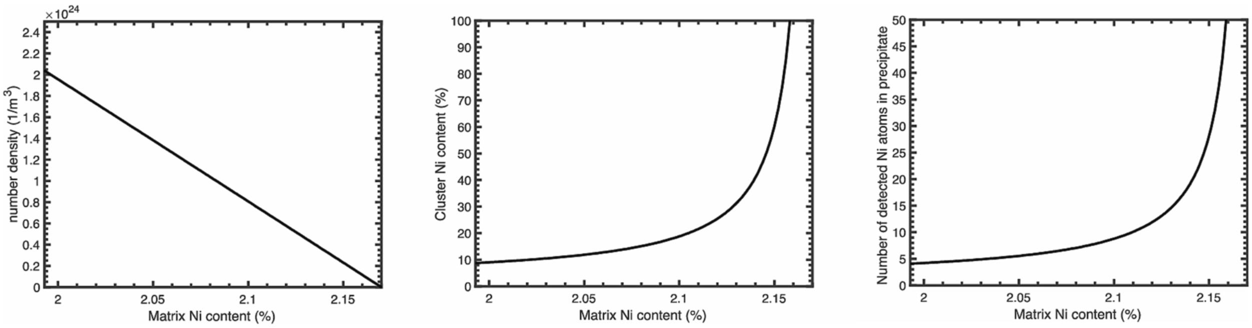

Fig. 3. Clustering characteristics of the decommissioned RPV, analysis 1, by the RDF clustering method [39]. The results are shown for different assumed matrix Ni contents. The total Ni content of this analysis is 2.17 at.% when the 29 peak is assigned as Ni only for the RDF.

Ni gives a number density of 5.2 10^{23} /m^3, 19 Ni atoms per cluster, and 41 at.% Ni in the clusters. This is possibly a slight overestimation, as some of the contribution in the RDF stems from effects from the field evaporation during analysis (compare with the reference material RDFs).

5. Conclusions

In this paper, the nanoscale structure of the unique weld metal of a decommissioned BWR RPV was investigated using APT. It was found that:

- There are no well-defined clusters of Ni, Mn, Si, and Cu in the material.
- However, statistical techniques identified a larger tendency of Ni atoms to be closer to each other than other atoms in the Barsebäck RPV material than in reference material that has not been exposed to neutron irradiation.
- Cu atoms also tend to be closer to Ni in the irradiated material. The trend for Mn and Si is less clear, but they are closer to Ni atoms in some analyses.
- The tendencies revealed using RDF statistical analysis can be interpreted as early stage clustering.

CRediT authorship contribution statement

Kristina Lindgren: Investigation, Formal analysis, Visualization, Conceptualization, Writing – original draft, Writing – review & editing, Funding acquisition. **Pal Efsing:** Conceptualization, Funding acquisition, Writing – review & editing. **Mattias Thuvander:** Conceptualization, Supervision, Funding acquisition, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.nme.2023.101466.

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