Atom probe study of irradiation-enhanced $\alpha'$ precipitation in neutron-irradiated Fe–Cr model alloys

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Abstract
Atom probe tomography (APT) was performed to study the effects of Cr concentrations, irradiation doses and irradiation temperatures on $\alpha'$ phase formation in Fe–Cr model alloys (10–16 at.%) irradiated at 300 and 450 °C to 0.01, 0.1 and 1 dpa. For 1 dpa specimens, $\alpha'$ precipitates with an average radius of 1.0–1.3 nm were observed. The precipitate density varied significantly from $1.1 \times 10^{23}$ to $2.7 \times 10^{24}$ 1/m$^3$, depending on Cr concentrations and irradiation temperatures. The volume fraction of $\alpha'$ phase in 1 dpa specimens qualitatively agreed with the phase diagram prediction. For 0.01 dpa and 0.1 dpa, frequency distribution analysis detected slight Cr segregation in high-Cr specimens, but not in Fe–10Cr specimens. Proximity histogram analysis showed that the radial Cr concentration was highest at the center of $\alpha'$ precipitates. For most precipitates, the Cr contents were significantly lower than that predicted by the phase diagram. The Cr concentration at precipitate center increased with increasing precipitate size.

1. Introduction
Ferritic–martensitic (F/M) steels are considered as lead candidate materials for Gen IV fission reactors and fusion reactors due to their superior swelling resistance, higher thermal conductivity, lower thermal expansion and adequate mechanical properties [1,2]. However, F/M steels exhibit low-temperature irradiation-induced embrittlement, which poses problems for their use below 400 °C [3]. In addition to the irradiation temperature limitations, Cr contents (2–18%) have been shown to substantially affect the irradiation-induced embrittlement and hardening [4,5]. The formation of dislocation loops and the Cr-rich $\alpha'$ precipitates under certain irradiation conditions is partly responsible for the embrittlement process.

Fe–Cr model alloys have been used to study the irradiation effects in F/M steels for decades. Earlier transmission electron microscopy (TEM) investigations on bcc Fe–Cr model alloys irradiated with heavy-ions and neutrons have considerably extended the understanding of the effects of Cr concentration and irradiation temperatures on the formation of the irradiation-induced dislocation loops [5–10].

$\alpha'$ precipitates are Cr-rich bcc phase coherent to the Fe-rich $\alpha$ matrix. TEM is not optimal for the study of $\alpha'$ precipitates because of the small misfit in lattice parameter between Fe-rich $\alpha$ and Cr-rich $\alpha'$ phases and the small difference in atomic mass between Fe and Cr atoms [11]. Instead, atom probe tomography (APT) has been shown to be the best tool for the characterization of these precipitates. Earlier APT studies focused on the aging effects in Fe–Cr alloys with higher Cr concentrations (>20%) [11–15]. However, the Cr concentration of engineering importance for nuclear structure materials is 9–12%, and the APT studies for this range have been lacking [16,17].

In order to evaluate the performance of F/M steels, a systematic understanding of the irradiation effects as a function of Cr concentration, irradiation temperatures and irradiation doses is important. In this work, poly-crystalline and single-crystal model alloys were irradiated with neutrons at 300 and 450 °C to doses of 0.01, 0.1 and 1 dpa. The irradiated and unirradiated specimens were investigated with APT to study the irradiation-enhanced $\alpha'$ precipitation.

2. Materials and experimental procedure
The materials examined in this study were poly-crystalline and single-crystalline Fe–Cr model alloys with nominal Cr...
concentration of 10 at.%, and 14 at.% Cr, respectively. The polycrystalline specimens have an average grain size of 180 μm. The chemical composition was measured by Carpenter Technology Corporation as 0.023 C, 0.006 N, <0.02 Al, <0.02 Si, <0.009 P, 0.004 S, 0.95 Cr, <0.01 Mn, 0.019 Co, 0.009 Ni, <0.009 Cu, <0.006 Mo (at.%). The single-crystal specimens were made in Los Alamos National Laboratory by Czochralski growth process. During the growth process, Cr was lost through evaporation, resulting in a crystal with decreasing Cr concentration along its length. The specimens were taken from cross-sectional slices of the original bar material and, therefore, had different Cr concentrations. The Cr concentration was, however, homogeneous inside individual specimens. The impurity level of single-crystalline specimens was similar to polycrystalline specimens. The composition of single-crystalline specimens was measured with APT. Table 1 summarizes the Cr contents $x_\text{Cr}$ and the corresponding irradiation conditions of all specimens where the error was the statistical uncertainty estimated by the peak decomposition routine of IVAS™ software.

Table 1: Nominal irradiation conditions and measured Cr concentrations of Fe–Cr single crystals.

<table>
<thead>
<tr>
<th>Specimen label</th>
<th>Irradiation conditions</th>
<th>$x_\text{Cr}$ (at.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Poly-crystals</td>
<td>Fe10Cr-PC</td>
<td>300, 450 °C No irradiation, 0.01, 0.1, 1 dpa</td>
</tr>
<tr>
<td>Single-crystals</td>
<td>Fe13Cr-SC</td>
<td>1 dpa–450 °C</td>
</tr>
<tr>
<td></td>
<td>Fe16Cr-SC</td>
<td>1 dpa–300 °C</td>
</tr>
<tr>
<td></td>
<td>Fe14Cr-SC</td>
<td>0.1 dpa–450 °C</td>
</tr>
<tr>
<td></td>
<td>Fe10Cr-SC</td>
<td>0.1 dpa–300 °C</td>
</tr>
<tr>
<td></td>
<td>Fe14Cr-SC</td>
<td>0.01 dpa–450 °C</td>
</tr>
<tr>
<td></td>
<td>Fe14Cr-SC</td>
<td>0.01 dpa–300 °C</td>
</tr>
<tr>
<td></td>
<td>Fe14Cr-SC</td>
<td>No irradiation</td>
</tr>
</tbody>
</table>

The size, density and volume fraction of $\alpha'$ precipitates were obtained by measuring the precipitates defined by the isosurfaces of 20%Cr. The density $N_p$ was calculated according to Eq. (1) and (2) [22].

$$N_p = \frac{N_{as1} + 0.5 \times N_{as2}}{V}$$

(1)

$$V = \frac{N_{Fe,Cr} \times a^3 \times 0.5}{\text{(detection efficiency)}}$$

(2)

where $N_{as1}$ is the number of $\alpha'$ precipitates well inside the boundary of the reconstruction data. $N_{as2}$ is the number of $\alpha'$ precipitates located at the edge of the boundary. As an estimate, half of the precipitates at the edge of the boundary were assumed to contribute to the density. $V$ is the specimen volume estimated using lattice constant $a$ (2.87 Å [23]), total number of Fe and Cr ions, $N_{Fe,Cr}$, and the detection efficiency.

For the precipitates well inside the boundary of the reconstruction data, the sizes were clearly defined by the isosurfaces and could be readily obtained. For precipitates located at the edge of the boundary, however, their sizes were not obtainable. Therefore, the mean size of $\alpha'$ precipitates was calculated by considering only the precipitates well inside the boundary. The volume fraction of $\alpha'$ precipitates was obtained through dividing the total volume inside isosurfaces by the specimen volume $V$.

The frequency distribution analysis reveals the degree of Cr segregation in a statistical fashion. The descriptions of frequency distribution analysis in APT can be found in [19]. In this study, the Pearson coefficient, $\mu$, was used to quantify the deviation of the measured frequency distribution $e(n)$ from the binomial distribution $f(n)$.

$$\mu = \sqrt{\frac{\chi^2}{N + \chi^2}}$$

(3)

The $\chi^2$ was defined as

$$\chi^2 = \sum_{n=0}^{n_s} \frac{(e(n) - f(n))^2}{f(n)}$$

(4)

where $n_s$ is the block size. The Pearson coefficient $\mu$ ranges between 0 and 1, corresponding to random distribution and complete decomposition, respectively. Smaller block size is more effective for detecting small-scale composition variations. However, when the block size is too small, spatial information is lost as nearby atoms are assigned into different blocks during the sampling process [19,24]. Based on recent APT studies on similar materials [25,26], a block size of 100 atoms was selected in this work.

A proximity histogram, or proxigram, was used to study the Cr concentration profile along the phase boundary normal of $\alpha'$ precipitates. A comprehensive introduction of proxigram was given in [21]. Multiple precipitates of similar sizes were combined to plot the proxigram in this study for better statistical significance. Alternatively, the Cr concentration profile of a single $\alpha'$ precipitate could be acquired by the use of the region of interest (ROI) across the precipitate boundary. However, the boundary curvatures of the $\alpha'$ precipitates in this study were too large (due to the small sizes of 1–2 nm) for ROI method to appropriately accommodate [18].

4. Results

4.1. Isoconcentration surface analysis

For the specimens of a dose equal and less than 0.1 dpa, few $\alpha'$ precipitates were found by using isosurfaces of 20%Cr or lower. Contrarily, $\alpha'$ precipitates were observed in all of the 1 dpa specimens. Fig. 1 shows the 20%Cr isosurface and Fig. 2 shows the size
distribution within the 1 dpa specimens. Table 2 summarized the size $r_{a 0}$, density $N_{a 0}$ and volume fraction $f_{a 0}$ of $a_0$ precipitates.

The morphologies of $a_0$ precipitates in 1 dpa specimens varied with $x_{Cr}$ and $T_{irr}$. The size was larger in 300 °C than in 450 °C irradiated samples. On the other hand, size increases with $x_{Cr}$, but less significantly. Therefore, it is suggested that $r_{a 0}$ was controlled more by the $T_{irr}$ than by $x_{Cr}$ within the temperature and composition range of this study.

Both the $T_{irr}$ and $x_{Cr}$ substantially affected the $N_{a 0}$. Specimens with higher $x_{Cr}$ and lower $T_{irr}$ exhibited higher $N_{a 0}$. Since $r_{a 0}$ were close to 1 nm for all conditions, the volume $f_{a 0}$ is essentially determined by $N_{a 0}$. The 300 °C Fe16Cr-SC has the highest $f_{a 0}$, followed sequentially by 450 °C Fe13Cr-SC, 300 °C Fe10Cr-PC and 450 °C Fe10Cr-PC, which is qualitatively in accordance with the phase diagram prediction [27,28].

Fig. 1. The reconstructed APT images of the 20%Cr isosurfaces of Fe–Cr model alloys irradiated by neutrons to a dose of 1 dpa at 300 °C and 450 °C.

Fig. 2. The size distribution of $a_0$ precipitates in Fe–Cr model alloys irradiated to 1 dpa.

Table 2

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Irradiation condition</th>
<th>$N_{a 0}$ ($\times 10^{15}$ m$^{-2}$)</th>
<th>$r_{a 0}$ (nm)</th>
<th>$f_{a 0}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe10Cr-PC</td>
<td>300 °C-1 dpa</td>
<td>7.7</td>
<td>1.18 ± 0.32</td>
<td>0.66</td>
</tr>
<tr>
<td>Fe10Cr-PC</td>
<td>450 °C-1 dpa</td>
<td>1.1</td>
<td>1.00 ± 0.36</td>
<td>0.072</td>
</tr>
<tr>
<td>Fe16Cr-SC</td>
<td>300 °C-1 dpa</td>
<td>27</td>
<td>1.25 ± 0.47</td>
<td>3.2</td>
</tr>
<tr>
<td>Fe13Cr-SC</td>
<td>450 °C-1 dpa</td>
<td>13</td>
<td>1.08 ± 0.32</td>
<td>0.85</td>
</tr>
</tbody>
</table>
effects on the
= 9.65% at 300
r
or
= 300–325
r/C24
is limited.
= 9–10% irradiated at
r
and
precipitate size is discussed in the discussion session.
The implication of the core Cr concentration dependence on the
0.6 dpa to 1 dpa on the core Cr concentration was insignificant.
Fe9Cr [29], implying that the effect of irradiation doses from
1.1 nm was coherent with earlier APT investigations on 0.6 dpa/C24
Cr concentration (difference was observed between the two temperatures. The core
Fe10Cr-PC specimens irradiated at 300 and 450
Fig. 4.
The core Cr concentration at the center of
0 precipitates as a function of
precipitates as shown inFig. 3. In more detail,Fig. 4 shows the
the core of precipitates without forming a plateau.
The Cr concentrations at the core of precipitates increased with
precipitate sizes as shown in Fig. 3. In more detail, Fig. 4 shows the
core Cr concentration as a function of precipitate size in 1 dpa
Fe10Cr-PC specimens irradiated at 300 and 450 °C. No significant
difference was observed between the two temperatures. The core
Cr concentration (~55%) of precipitates with a size around
1.1 nm was coherent with earlier APT investigations on 0.6 dpa
Fe9Cr [29], implying that the effect of irradiation doses from
0.6 dpa to 1 dpa on the core Cr concentration was insignificant.
The implication of the core Cr concentration dependence on the
precipitate size is discussed in the discussion session.

4.3. Frequency distribution analysis

For specimens with an irradiation dose lower than 1 dpa, it is
difficult to use isosurface to reveal χ' precipitates. Instead,
frequency distribution analysis was found useful to study the early
stage of Cr segregations. The frequency distribution and the
corresponding binomial distribution of Fe–Cr specimens are shown
in Figs. 5–7. The μ-index derived from the frequency distribution is
shown in Fig. 8. As Fig. 5 shows, the frequency distributions of the
un-irradiated specimens are very close to the binomial distributions,
denoting negligible χ' phase in the as-received specimens.
Up to 0.1 dpa, the frequency distributions of irradiated Fe10Cr-
PC specimens are similar to as-received specimen. No evident
segregation of Cr atoms was detected. At 1 dpa, however, con-
siderable deviation, with frequencies moved toward two sides,
was observed as shown in Fig. 6. In addition, the deviation between
the observed distribution and the binomial distribution was higher
in 300 °C than in 450 °C irradiated samples.
For irradiated Fe–Cr single crystals (higher xCr), Cr segregation
increased with irradiation dose. Some Cr segregations at lower
doses of 0.01 and 0.1 dpa were detected, which was different from
Fe10Cr-PC specimens. This result indicated that the Cr segregations
began earlier in Fe–Cr alloys with higher xCr. Because of the varia-
tions in xCr, it was more difficult to evaluate the Tnir effects on the
Cr segregations in single-crystalline specimens. For instance, the
0.1 dpa Fe10Cr-SC specimen irradiated at 300 °C exhibited a
particularly low Cr content (10.02%), which resulted in lower degree
of Cr segregation.

5. Discussion

5.1. Effects of irradiation dose on the χ' precipitation

Precipitation kinetics of the χ’ phase is much faster under
irradiation than under thermal aging conditions alone due to
irradiation-enhanced diffusivity [15,30]. Fig. 9 shows a compilation
of experimental data showing the evolution of fχ’ for Fe–Cr alloys
with Cr contents xCr = 9–10% irradiated at Tirr = 300–325 °C. The
dashed line is the fχ’ for Fe–Cr with xCr = 9.65% at 300 °C predicted
by the phase diagram using lever rule [28]. Except for one data
point from [30], fχ’ remains below the saturation limit as it evolves
with irradiation. Provided the sensitivity of 20%Cr isosurface, a
threshold dose between 0.1 and 0.6 dpa was suggested for detect-
ing the formation of χ’ precipitates in Fe10Cr irradiated at ~300 °C.
Beyond 0.6 dpa, the fχ’ increases with dose and approaches the
saturation limit. On the other hand, the corresponding mean precipitate
size fχ’ (not shown) is within 1.0–1.3 nm, and there is no
evident trend of fχ’ with dose. Therefore, for this specific condition,
it is still in the nucleation regime. For other xCr, or Tnir, it may enter
growth regime at a lower dose. For instance, larger χ’ precipitates
with radius fχ’ = 2–5 nm were observed in Fe–Cr with xCr ~12% irradiated at 400 °C to a dose of ~7 dpa [9,31]. However, a systematic
investigation over a range of dose is currently not available
because the irradiation data for those xCr and Tnir is limited.

5.2. Effects of Cr concentration on the irradiation-enhanced χ’ precipitation

The χ’ precipitation in Fe–Cr alloys is significantly dependent on
xCr. Under equivalent irradiation condition, Fe–Cr alloys with
higher xCr have larger fχ’, higher precipitate density Nv, higher χ’
volume fraction fχ’, and lower threshold dose for observable
precipitation.
As shown in Table 2, fχ’ increases slightly with xCr, at both 300 °C and
450 °C. This is consistent with the Monte Carlo modeling on
thermally-aged bcc Fe–Cr [32,33]. Since the Cr diffusivity does
not increase with xCr, the growth rate of precipitates is not the
cause. Instead, the onset dose of nucleation accounts for this

Fig. 3.
The proxigrams to the 20%Cr isosurfaces of χ’ precipitates in Fe10Cr-PC specimens irradiated at 300 °C to 1 dpa. Three size groups are plotted: fχ’ = 1.5–1.55 nm, 1.3–1.42 nm and 0.7–0.85 nm.

Fig. 4.
The core Cr concentration at the center of χ’ precipitates as a function of precipitate size in radius in 1 dpa Fe10Cr-PC specimens irradiated at 300 and 450 °C.
phenomenon. The precipitates in specimens with higher \( x_{cr} \) nucleate \( \alpha' \) precipitates earlier and they have more time to grow. This is supported by the frequency analyses in this study, and by modeling [33]. Nevertheless, the \( T_{irr} \) is relatively insensitive to \( x_{cr} \), consistent with other experiments and modeling [16,29,30,32,33].

On the other hand, \( N_x \) increases significantly with \( x_{cr} \). This can be explained using the Gibb’s homogeneous nucleation theory, expressed in Eq. (5) [34],

\[
N_x \propto x_{cr} \left( \frac{\Delta G^*}{k_{RT}} \right)
\]  

\( \Delta G^* \) is the activation energy for \( \alpha' \rightarrow \alpha' \) phase transformation, \( k \) is gas constant, and \( T \) is absolute temperature. \( \Delta G^* \) decreases with \( x_{cr} \) because of the increasing driving force of phase transformation with \( x_{cr} \). Since both \( x_{cr} \) and \( \Delta G^* \) increases with \( x_{cr} \), \( N_x \). The observation of this study that \( N_x \), and not \( \tau_{cr} \), varies strongly with \( x_{cr} \) is in accordance with the modeling result in [33].

To study the \( x_{cr} \) effects on \( f_{irr} \), a compilation of experimental data is shown in Fig. 10 for Fe–Cr ferritic alloys irradiated at about 300 °C to a dose around 1 dpa. The dashed line is the \( \alpha' \) volume fraction predicted by the phase diagram in Ref. [28] using lever rule at 300 °C. No \( \alpha' \) precipitation was observed below the threshold \( x_{cr} \), of 8.1%, which is consistent with the prediction from the phase diagram. Beyond the threshold, \( f_{irr} \) increases with \( x_{cr} \). As discussed above, this is primarily due to the higher nucleation rate \( N_x \) in the specimens with higher \( x_{cr} \).

It needs to be mentioned that the comparisons in Figs. 9 and 10 involve uncertainties. The reported volume fractions were based on varied techniques where each technique has its own limitations and uncertainties. For instance, SANS does not detect cluster size smaller than 0.5 nm, resulting in a slight underestimation of volume fraction [35]. For APT, the trajectory aberration and local magnification effects can slightly change the obtained size of \( \alpha' \) precipitates [20]. In addition, the use of isosurface also introduces uncertainties because the apparent precipitate size and volume fraction depend on the Cr concentration chosen to define the isosurface, while the interface of \( \alpha' \) precipitates is not well defined.

5.3. Irradiation temperature effects on the irradiation-enhanced \( \alpha' \) precipitation

As shown in Fig. 1 and in Table 2, \( T_{irr} \), \( N_x \) and \( f_{irr} \) of \( \alpha' \) precipitate in Fe–Cr specimens decrease with \( T_{irr} \). This is in agreement with the prediction from the phase diagram in [28] that \( f_{irr} \) decreases with increasing \( T_{irr} \). The observation of lower \( N_x \) with higher \( T_{irr} \) might be attributed to a decreasing nucleation rate \( N_x \) with increasing \( T_{irr} \). In terms of Gibb’s homogeneous nucleation theory in Eq. (5) [34], \( T_{irr} \) has effects on both the \( \Delta G^* \) and the \( RT \) terms. When \( T_{irr} \) increases, the \( RT \) terms result in an increase in \( N_x \). On the other hand, increasing \( T_{irr} \) results in a reduction in the driving force for precipitation, leading to an increase in \( \Delta G^* \), and therefore, and a decrease in \( N_x \). In this case, the effect of increasing \( T_{irr} \) on the \( \Delta G^* \) term is inferred to be more significant than on \( RT \) term.

The size \( \tau_{cr} \) decreases with increasing \( T_{irr} \). Compared to \( x_{cr} \), the effect of \( T_{irr} \) on \( \tau_{cr} \) is more significant. The Cr diffusivity is not applicable to interpret this temperature dependence because increasing \( T_{irr} \) should enhance Cr diffusivity and the precipitate growth rate, provided diffusion-controlled mechanism. Therefore, there must be other mechanism controlling the growth of \( \alpha' \) precipitate. Perhaps, the smaller \( \tau_{cr} \) with \( T_{irr} \) is due to the smaller \( N_x \) that come with a delayed onset for precipitation growth. These questions remain unclear at this point.

5.4. The core Cr concentration dependence on the size of \( \alpha' \) precipitates

As Fig. 4 shows, the majority of precipitates in this study had a core Cr concentration considerably below the equilibrium
concentration predicted by the phase diagram (>90%). Several hypotheses have been proposed to explain this APT observation, such as the non-classical nucleation, the modified phase diagram and APT artifacts. The non-classical nucleation explanation is not favored due to the low nominal Cr contents in this study [14,16]. The irradiation-induced point defects (super-saturated) have been

Fig. 7. Frequency distribution analysis for single-crystalline specimens irradiated at 300 °C to a dose of (a) 1 dpa and (c) 0.1 dpa (e) 0.01 dpa, and at 450 °C to the doses of (b) 1 dpa and (d) 0.1 dpa (f) 0.01 dpa. The bin size $n_b$ is 100.

Fig. 8. The $\mu$-index of un-irradiated and irradiated (a) poly-crystalline Fe10Cr and (b) single-crystalline specimens, based on the frequency distribution analysis.
suggested to change the phase diagram, resulting in a higher Fe solubility in Cr [15,16]. However, this cannot explain the observed correlation between the core Cr concentration and precipitate sizes.

The blurring of the APT data at the interface could cause a deficit of precipitate component (Cr for this study) in very small clusters [21]. As the size of the precipitate increased, the blurring effects at the interface became less significant and therefore the core Cr concentration would approach the equilibrium value. This hypothesis is more in agreement with the concentration-size correlation. Finally, another plausible explanation from the work of Svetukhin et al. showed that the precipitate composition is determined by the interface surface energy in the Fe–Cr binary system, resulting in increasing Cr content of $\alpha'$ precipitates with increasing precipitate size [36]. To justify these hypotheses, further studies combining experiments, atom probe simulation and thermodynamic calculations are required.

6. Conclusion

Ferritic Fe–Cr model alloys have been irradiated with neutrons at 300 and 450 °C to the doses of 0.01, 0.1 and 1 dpa. Atom probe tomography has been performed to investigate the $\alpha'$ decompositions in those specimens. The $\alpha'$ precipitates of size of 1–2 nm appeared in all of the 1 dpa specimens. The relative volume fraction between individual specimens was consistent with the prediction from the phase diagram. The density of $\alpha'$ precipitates increases significantly with increasing Cr content and decreasing irradiation temperature. The size of $\alpha'$, however, is relatively insensitive to the Cr content and the irradiation temperature.

In Fe–10Cr poly-crystals of doses equal and less than 0.1 dpa, no Cr segregation could be detected with the analysis techniques employed in this study. On the other hand, some increases in Cr segregation were observed in single-crystalline specimens (with higher Cr content) of equivalent dose by the use of frequency distribution analysis.

The Cr concentration profiles of the $\alpha'$ precipitates in the 1 dpa specimens were examined using x-ray analysis. The profiles were similar for all of the conditions; i.e. Cr concentration increased toward the precipitate core without forming a plateau. In addition, it has been shown that core Cr concentration increased with precipitate size.

This work provides experimental information on the microstructure evolution of Fe–Cr model alloys under neutron irradiation in atomic scale as a function of Cr concentrations and irradiation temperatures. The results are useful for understanding the mechanical property degradation of F/M steels, and could serve as the benchmark for atomistic simulations in this class of materials.

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