# Experimental report

13/06/2016

**Proposal:** **1-04-88**

**Council:** 10/2014

**Title:** Temporal evolution of Cu segregation in binary Fe-Cu alloys: A comparative study between thermal aging and ion irradiation

**Research area:** Materials

**This proposal is a new proposal**


**Main proposer:** **Aniruddha BISWAS**

**Experimental team:** Sudip Kumar SARKAR
Aniruddha BISWAS

**Local contacts:** Dirk HONECKER


**Samples:** Fe-Cu

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| D33 | 3 | 0 | | |
| D22 | 3 | 1 | 14/07/2015 | 15/07/2015 |
| D11 | 3 | 0 | | |


## Abstract:

Fe-Cu binary alloy is an appropriate prototype for reactor pressure vessel steels (RPV), since segregation of Cu is one the main reasons for embrittlement in RPV steels under irradiation. Interestingly, long-term thermal aging also can emulate this radiation-induced segregation. For a comparative study, a series of Fe-Cu alloys (0.1–1.4 at.% Cu) were subjected to: (i) Aging at 773 K for: 1, 3, 5, 8, 25, 50 and 100 h; and (ii) Irradiation up to 1 dpa with 8 MeV proton beam. Segregation starts as fine Cu-rich coherent spheres that evolves into cylinders beyond a threshold size and small-angle neutron scattering (SANS) is especially suited to study this phenomenon along with its complex transition. The current study will explore the temporal evolution of segregation by combining ex-situ SANS with complementary techniques 3DAP & TEM, followed by in-situ SANS at 773 K. Combination of these complementary techniques would provide comparative perspectives and valuable insight into evolution of solute segregation process.

## Experimental report (1-04-88)

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**Temporal evolution of Cu segregation in binary Fe-Cu alloy**: Due to the low solubility limit of Cu in Fe, long term thermal aging leads to Cu segregation, which is quite complex due to its association with changes in crystal structure, chemical composition, interface character, morphology and size. Small angle neutron scattering is suited for these kinds of studies since the mesoscopic density fluctuation due to segregation of Cu can be probed by this technique. The current study explores the quantitative and qualitative information on the Cu segregation in combination with TEM, magnetization study and hardness study.

DSC analysis manifests the segregation behavior due to heating (Fig.1a). Fe-Cu shows bcc microstructure at room temperature for all aging time because of low volume fraction of precipitate (fig.1b). Bright field TEM micrographs (fig.1c) display the Cu precipitates after aging of Fe-0.35 at% Cu alloy for 2 different durations. Typical rod-shaped Cu precipitates are observed after aging for 100 h, as evidenced in the atom map obtained from 3DAP microscope (Fig.2a). Hardness plot with aging time for all four different compositions is displayed in fig.2b. Magnetization plot with aging time is displayed in fig.2c.

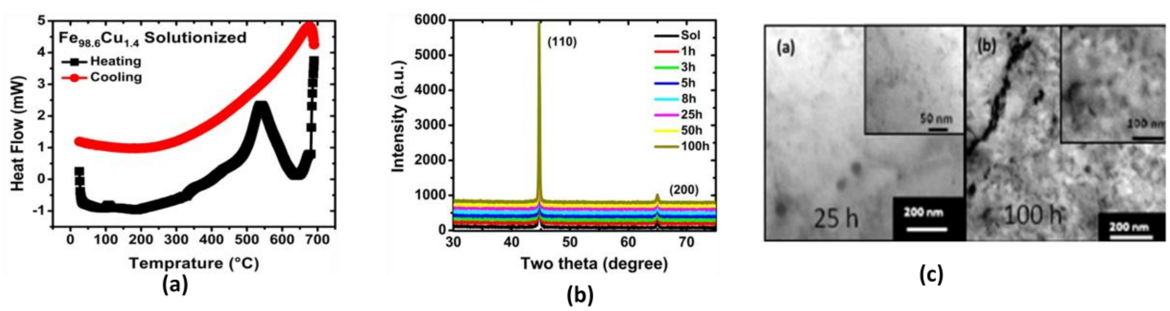


*__Fig.1:__ (a) DSC plot of solutionized sample (b) XRD pattern (C) TEM micrographs for 25h & 100h*

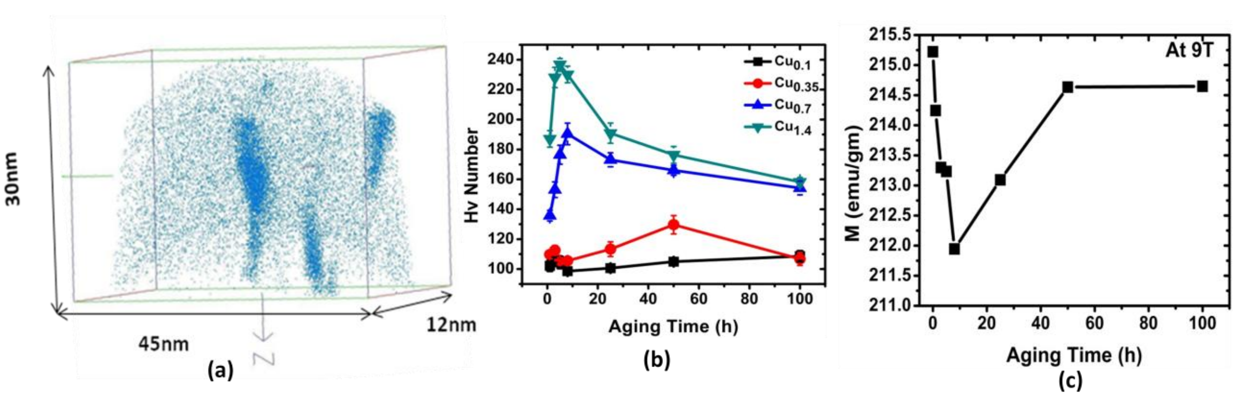


*__Fig.2__: (a) 3DAP atom map showing Cu segregation for 100h (b) Hardness plot for all composition (C) Magnetization plot for FeCu1.4*

TEM micrographs and 3DAP atom map are direct evidence of Cu segregation in thermally aged samples. In order to correlate microstructural change with bulk properties, we have carried out hardness and magnetization studies with aging time. Hardness curve first increase up to certain aging time and then decrease. Magnetization curve behave reciprocal to the hardness curve because domain wall motion is hindered by Cu precipitates up to certain size.

SANS experiments were carried out at ILL, Grenoble using neutrons to study this segregation. Our SANS data show gradual shift towards lower q value with increasing aging time, signifying increase in precipitate size. The data corresponding to thermal aging experiments are fitted for spherical particles and are plotted in Fig. 3(a). The radius vs aging time plot is modeled with a linear fit that shows the growth of median radius to follow a time$^{1/2}$ power law (fig.3(b)). The SANS data for the irradiated samples are also fitted for spherical particles (fig. 3(C)), where the median radius is found to decrease with increasing Cu content (table1). Table 1 and 2 below show the extracted parameters. The detailed study is underway.

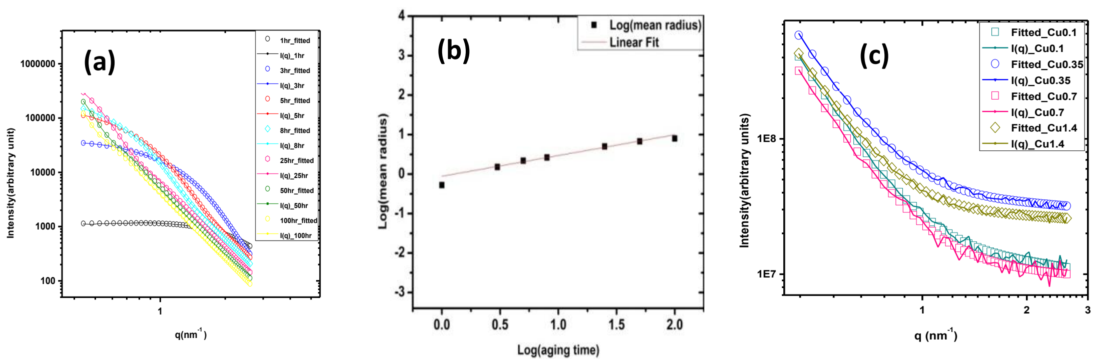


***Fig 3:*** *(a) SANS fitted profile for thermally aged samples; (b) Variation of Cu precipitate with aging time; (c) SANS fitted profile for irradiated samples*

| **Cu content** | **Mean Radius(nm)** | **Polydispersity** |
|---|---|---|
| 0.1 | 0.27 | 0.2 |
| 0.35 | 0.19 | 0.2 |
| 0.7 | 0.14 | 0.2 |
| 1.4 | 0.13 | 0.2 |

**Table 1**: Extracted Cu precipitate information from SANS data for irradiated samples

| Aging Time(h) | Mean Radius(nm) | Error in mean radius(nm) | polydispersity |
|---|---|---|---|
| 1 | 0.52 | 0.0055 | 0.4 |
| 3 | 1.52 | 0.00058 | 0.24 |
| 5 | 2.21 | 0.00068 | 0.27 |
| 8 | 2.68 | 0.0007 | 0.27 |
| 25 | 5.04 | 0.0011 | 0.26 |
| 50 | 6.78 | 0.0014 | 0.25 |
| 100 | 7.86 | 0.0023 | 0.24 |

**Table 2**: Extracted Cu precipitate information from SANS data for thermally aged sample