Study of Effect of Mode of Cooling on U Trace Contents in Annealed Copper Samples Using Solid State Nuclear Track Detectors (SSNTDs)

Satish Kumar Garg
Govt. P G College Ambala Cantt
(Haryana) India

Abstract
With the change of concentration of the trace elements, changes in physical, mechanical and chemical properties of materials also occur. Metals having high concentration of Uranium(U) are expected to suffer radiation damages caused by the fission fragments which change the physical properties of these metals. In the present work, U has been estimated in various samples of Cu. These Cu samples were heated/annealed to different temperatures for different time intervals consequently cooled under different conditions. U trace content in Cu was found to be more in water quenched samples than in air cooled samples annealed to the same range of temperature for the same period of time.

Index Terms: U Trace Contents, Particle Analysis Technique, SSNTDS, Air Cooled and Water Quenched Cu Samples

IIntroduction
Elements whose concentration is very small (of the order of ppm) as compared to bulk of the material are referred to as ‘Trace Elements’. With the change of concentration of these trace elements, changes in physical, mechanical and chemical properties of materials also occur. Metals having high concentration of uranium are expected to suffer radiation damages caused by the fission fragments which change the physical properties of these metals. In addition to the usual radiation damages, α-particles are also emitted in the decay process of Uranium. As little as 10⁻⁸ atomic fraction of Helium causes an appreciable decrease in the equi-cohesive temperature in the reactors [1]. Thus low U content metals are desired for fabrication of rector and allied structures. At the same time excess presence of U in utensils causes health hazards because U is an α-particle emitter. It, being non-volatile at the temperature of cooking, could be a potential cause of bladder and bronchial lung cancer as it could enter the bronchie and get accumulated in bronchial bifurcations. The recent findings are...
that drinking of tea brewed in aluminium utensils can adversely affect the functioning of brain. It is a matter of special concern to all of us. Hence there is a need for its estimation in metals.

2 Experimental

2.1 Material

In the present work, U has been estimated in various samples of Cu. Cu is being selected because they are used in engineering field. While in use Cu is often exposed to very high temperature for long durations.

To estimate the concentration of U in Cu Lexan as Solid State Nuclear Track Detectors (SSNTDs) has been used.

2.2 Technique

Various techniques like emission spectrometry, mass spectrometry, X-ray spectrometry, radio tracer method, neutron activation analysis etc. can be used to estimate trace elements in copper, but they require expensive equipment, large quantity of sample, have contamination problem etc. whereas particle analysis technique has been selected to estimate U in Cu because of the following reasons:

1. It does not involve complicated and costly electronic circuits.
2. Particles leave their permanent impression in the detectors which can be studied at the convenient time.
3. It is very economical and rapid.
4. It can record a level of concentration than is possible by any other method.
5. It enables micro-mapping of the trace content in a region of the material which is not possible by any other technique.
6. It involves least storage problem at ambient temperature.

This technique is based upon the principle that charged particles while passing through an insulator destroy the local order in crystalline matrix. These effects are called radiation damages and are stored in the solids, and later revealed by chemical etching.

2.3 Sample Preparation

The samples of copper were taken in the form of rods, cut into discs having diameter 1.2 cm and thickness of 2 mm. A usual approach is to grind these samples on rotating cast iron lap with 200, 400 and 600 mesh carborundum powders and finally polish with diamond pastes of 8, 6, and 0.25 microns to have a good contact with Lexan detectors. These copper samples were heated/annealed to 300, 450 and 600 °C different for 4, 10 and 15 hr. Circular discs of Lexan plastic, having diameter 1.2 cm served as detectors for recording the fission tracks. Copper samples, after heat treatment and consequently cooling under different conditions, were covered with detectors and tightly packed in an aluminium capsule. The enclosed samples were irradiated to a known dose of thermal neutrons at CIRUS reactor at BARC Trombay, Bombay. After irradiation, detectors were etched in 6.25 N NaOH solution at 70 °C and scanned under optical microscope for track counting.

3 Results And Discussions

The following results are obtained:

1. U trace contents in Cu at room temperature was found to be 1 ppm.
2. U trace contents in Cu samples annealed at 300 °C for a period of 4, 10 and 15 hr and subsequently cooled in air and quenched in water samples have been given in Table 1.
3. No U trace content was obtained in Cu samples annealed at 450 and 600 °C for a period of 4, 10
and 15 hr in air cooled and water quenched samples.

The results are plotted in Figure 1. From the Table 1 and Figure 1, following conclusions are drawn:

1. The uranium trace content in copper initially increases on annealing but decreases when annealed for longer time intervals (10,15 hr) or to higher range of temperature of the order of 450 and 600 °C.

2. The U trace content in Cu is more in water quenched samples than in air cooled samples annealed to the same range of temperature for the same period of time.

This initial increase in the uranium content can be explained if we consider that the diffusion rate of uranium from within the matrix onto the surface is more than the diffusion rate of uranium from surface into the matrix on account of heating resulting in a net increase in uranium content on the surface. In the present study, copper is the host matrix and uranium is the impurity atom and diffusion coefficient of uranium in copper is small. This fact can be stated on the basis that diffusion coefficient of impurity atoms decreases as the atomic number of the impurity atom increases[2]. At the same, diffusion coefficient increases with rise in temperature [3]. When samples are heated for longer duration of time or to higher range of temperature, the net diffusion of U is into the matrix i.e., from region of higher concentration (surface of the samples) to region of low concentration (inner matrix).

The decrease in air cooled and water quenched samples has been observed to be almost exponential.

### Table 1: U trace content for Air Cooled and Water Quenched Cu Samples

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>Period of Heating (hr)</th>
<th>U content for Air Cooled Samples (ppm)</th>
<th>U content for Water Quenched Samples (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>4</td>
<td>2.1</td>
<td>2.8</td>
</tr>
<tr>
<td>2.</td>
<td>10</td>
<td>0.5</td>
<td>1.4</td>
</tr>
<tr>
<td>3.</td>
<td>15</td>
<td>0.3</td>
<td>0.9</td>
</tr>
</tbody>
</table>

The increase in uranium content in water quenched samples as compared to air cooled samples can be explained on the basis that quenching of samples in water abruptly stops the motion of impurity atoms. Thus the system freezes in the same state that was present at the time of quenching. But in air
cooled samples, decrease in uranium content is observed which depends upon time of heating of the samples. This could be due to the net diffusion of uranium into the matrix. The other reason could be due to formation of uranium oxide compound in presence of air which is a stoichiometric compound. The process of oxidation is accelerated due to copper which acts as catalyst[4]. There is every possibility that some amount of this compound is lost at the time of processing of the samples. The net amount of U which disappears from the surface of the air cooled samples has been found to be more or less uniform for different periods of heating. For samples heated at 300°C, the amount of U lost is 0.7, 0.9 and 0.6 ppm for samples annealed at 4, 10 and 15 hr respectively. These results lead to the conclusion that amount of U decreased in the form of Uranium dioxide is nearly independent of the period of heating keeping the temperature constant. In case of the samples which are heated upto 600 °C for different periods of times like 4,10 and 15 hr, no U content was found on the surface of these samples. It can be probably due the reason that at 600 °C, net diffusion of U into the matrix increases due to increase in the diffusion coefficient. Hence no significant amount of U was found on the surface of the samples.

4 Conclusions

From the Table and Figure, following conclusions are drawn:

(1) The uranium trace content in copper initially increases on annealing but decreases when annealed for longer time intervals (10,15 hr) or to higher range of temperature of the order of 450 and 600 °C.

(2) The U trace content in Cu is more in water quenched samples than in air cooled samples annealed to the same range of temperature for the same period of time.

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5 References