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# Using Sodium as Filling and Heat-Conducting Material in the Irradiation Capsules of the Fusion Materials Irradiation Facility IFMIF-DONES

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#### Abstract

The capsules of the IFMIF-DONES High Flux Test Module (HFTM) are packed densely with Eurofer specimens. A filling material (previously NaK-78 and presently sodium) is needed to fill any empty volume to improve the heat conduction and obtain uniform temperature distribution. NaK-78 is replaced by Sodium because potassium generates argon isotopes leading to a pressure increase and formation of bubbles which counteract the purpose of introducing NaK-78 as a heat conductor. In this experimental study, two setups are used to investigate sodium as the new filling material. The first setup is dedicated to test the wettability of various specimens of Eurofer and stainless steel 316L by liquid sodium within the temperature range of 100°C to 430°C. The second setup consists of: (i) a full scale prototype of the HFTM capsule packed with Eurofer specimens, (ii) a stainless steel container for melting the sodium, and (iii) heaters, thermocouples and supporting parts. The objective is to demonstrate successful sodium filling and emptying of the prototype capsule and to determine how well the specimens are wetted and the empty volumes are filled with sodium. All experiments are performed inside a glovebox filled with dry argon to maintain an oxygen- and moisture-free atmosphere. The results of these experiments, which are relevant to IFMIF-DONES HFTM capsules and other sodium-using applications, are presented and discussed in this paper. For example, the results showed that efficient wettability of Eurofer and stainless steel 316L specimens by sodium may be assured when the temperature of the steel-sodium system is 430°C or higher.

Keywords: fusion, sodium, Eurofer, irradiation, IFMIF-DONES

#### 1. Motivation and Objective

The High Flux Test Module (HFTM) is considered the main module in the fusion facility IFMIF-DONES (International Fusion Materials Irradiation Facility- Demo Oriented NEutron Source). The HFTM capsules are packed densely with Eurofer specimens and it was proposed to use the sodium-potassium eutectic alloy (NaK-78) to fill the gaps among these specimens and any empty volume in order to improve the heat conduction and obtain uniform temperature distribution. A previous experimental study [1] demonstrated that the Eurofer specimens are wetted efficiently by NaK-78. In addition, another study [2] investigated the chemical compatibility between NaK-78 and the Eurofer specimens. However, the relevant nuclear calculations recently showed that the presence of potassium in NaK-78 leads to generation of several argon isotopes, most of them are radioactive. The generated amount of argon is rather high, several ccm (at Standard Temperature and Pressure) and would lead to a pressure increase ~30 bar in the capsule. Furthermore, argon which does not dissolve in the NaK-78 will likely appear as dispersed bubbles inside the capsule and possibly rise partially to the expansion volume. This, unfortunately, counteracts the purpose of introducing NaK-78 as a heat conductor; therefore, a new filling material is needed. One option could be pure sodium which does not produce argon or other insoluble gases. But because the melting temperature of sodium is higher than the room temperature and higher surface tension, filling and wetting processes need to be re-demonstrated. Accordingly, this experimental study aims to: (i) test the wettability of Eurofer and AISI 316L stainless steel specimens by sodium within the temperature range (100°C to 430°C), and (ii) demonstrate a successful sodium filling process using a prototype capsule in order to have a better understanding of how well the specimens are wetted and the gaps are filled with sodium.

#### 2. Introducing Sodium

Sodium has been used in the chemical industries and energy field in addition to the scientific research. For nuclear energy, liquid sodium has been used as a coolant for the sodium-cooled fast reactor due to its relevant properties. Studying the compatibility of sodium with structural materials and its operational safety-related issues started a long time ago (since 1950's) for the development of the sodium-cooled nuclear reactors [3]. So, there is an extensive experience and significant developments have been dedicated for using sodium in the nuclear energy field. Sodium is a soft ductile alkali metal with shiny silvery color. It is opaque and solid at room temperature. Oxygen reacts with sodium and produces high-density white fumes composed of sodium oxides. Also, sodium reacts with water (even moisture in the air) exothermically to produce flammable hydrogen gas and sodium hydroxide leading to hazards of fire and explosion. Sodium melts at 98°C and boils at 882°C; hence it maintains its liquid phase over an advantageous wide range of temperature. In addition, sodium has several advantages which are reported in reference [4] as follows. First, sodium has good heat capacity and thermal conductivity that allows heat to be transferred efficiently. Second, pure sodium has very good

compatibility (i.e. practically little corrosive) with steels. It is worth to mention that purification of sodium has been efficient and well developed. Third, sodium barely absorbs neutrons as it has a small neutron capture cross section. Irradiation of nuclear-grade sodium by neutrons produces two radioactive isotopes: <sup>24</sup>Na (half-life: 15 h) and <sup>22</sup>Na (half-life: 2.6 year). Furthermore, sodium features good wetting characteristics [5] due to the possibility of reducing its oxygen content to less than 3 ppm. However, there are some difficulties of using sodium. First, sodium is highly reactive with water and oxygen producing hazardous by-products, such as hydrogen or caustic substances and may lead to a sodium fire or explosion. Therefore sodium should always be handled, transferred and stored under an inert atmosphere and the necessary safety measures must be taken to avoid any sodium leakage to the air. Second, sodium is solid below 98°C therefore to drain sodium from the HFTM capsules heating above 98°C is required. This is one of the main differences between sodium and NaK-78 where the last is liquid at room temperature. Third, treatment of sodium and involved components after testing needs special precautions and efforts.

#### **3. Experimental Work**

The main experimental activity is to achieve an effective filling of the HFTM capsule with liquid sodium; nevertheless some wettability tests are performed first in order to determine the temperatures that produce an efficient wetting of the Eurofer specimens.

#### 3.1 Wettability Testing

The wetting ability (referred as wettability) depends on several factors such as surface characteristics and conditions, temperature, and purity of sodium. The sodium, used in the current experiments, has a purity of 99.8%. Undoubtedly temperatures of both the material surface and liquid sodium have a significant effect on the wetting process. An experimental setup was constructed at KIT to test the wettability of Eurofer and AISI 316L stainless steel specimens by sodium within the temperature range (100°C to 430°C). This setup, shown in Figure 1, consists of: (i) a container made of AISI 316L stainless steel, (ii) two parallel plates, (iii) four cartridge heaters, (iv) four thermocouples, (v) metallic ruler, and (vi) various supporting parts. The container has outer dimensions of  $190 \times 90 \times 40 \text{ mm}^3$  and its cavity (to contain sodium) has dimensions of  $80 \times 80 \times 20 \text{ mm}^3$ . The two parallel plates are aligned perpendicularly to the free surface of sodium in the container cavity. The plates are clamped together and closely spaced with a spacer between them using two screws in order to form a parallel-plates channel. The channel thickness was adjusted to 0.3 mm by inserting a steel spacer with dimensions of  $30 \times 25$ 

mm<sup>2</sup>. For more details about this setup and the experimental procedures, see reference [1]. All experiments were performed inside a dedicated glove box filled with dry argon to maintain an oxygen- and moisture-free atmosphere where both the oxygen and water amounts are always less than 0.1 ppm. Due to wettability and capillarity, the liquid sodium may rise in the gap between the parallel plates over its level in the free surface. The height of the sodium capillary rise is recorded using a digital camera and then is determined with the help of the metallic ruler located next to the parallel plates. The capillary rise of sodium in the channel is used as an indication measure of how well the wettability is.



Figure 1: Setup of wettability testing.

#### **3.1.1 Wettability of Parallel Plates**

In this section, the results of three successful wettability runs are presented. For each run, the material, surface roughness, and manufacturing technique of the parallel plates are given. The surface roughness is expressed in terms of the parameter  $R_a$  (its value is given in  $\mu$ m). In addition, the parallel plates are identified by numbers 1 to 6. The results of the first experimental run are shown in Figure 2 where the wetting of plates 1 and 2 and the sodium rise between the plates at 420°C and 430°C are observed. The sodium rise can be seen as a shiny silver line between the dark plates. Plate 1 ( $R_a = 0.30$ ) and plate 2 ( $R_a = 0.32$ ) were surface finished by spark erosion and their material is Eurofer.



Figure 2: Sodium rise at 420°C and 430°C for Eurofer plates 1 & 2.



Figure 3: Sodium rise at 420°C and 430°C for Eurofer plates 3 & 4.



Figure 4: Sodium rise at 420°C and 430°C for 316L SS plates 5 & 6.

Similarly the wetting and sodium rise for plates 3 and 4 are presented in Figure 3. Plate 3 ( $R_a = 0.70$ ) and plate 4 ( $R_a = 0.83$ ) were surface finished by grinding and their material is Eurofer. The third wetting run is given in Figure 4 where plates 5 and 6 are tested. Plate 5 ( $R_a = 1.16$ ) and plate 6 ( $R_a = 0.69$ ) were surface finished by milling and their material is AISI 316L stainless steel. Wettability of the fusion-relevant steels (Eurofer and AISI 316L stainless steel) by liquid sodium was experimentally tested within the temperature range (100°C to 430°C). For both the tested

Eurofer pairs and also the AISI 316 stainless steel, similar results were observed as follows: (i) the liquid sodium does not wet the steel surface within the temperature range of 100°C to 380°C, (ii) approaching the temperature of 390°C, the sodium starts to wet the side surfaces of the parallel plates, (iii) reaching the temperature of about 415°C, sodium starts to make a complete wetting of the plates surfaces and also rise in the channel between the plates, (iv) between 420°C to 430°C, the sodium rise increases with temperature. From the obtained results, it is concluded that wettability of Eurofer and 316L stainless steel by sodium under IFMIF-relevant machining and surface roughness can be assured when the temperature of the steel-sodium system is 430°C or higher. In the previous study [1] of wettability of Eurofer and 316L stainless steel by NaK-78, the temperature of 350°C is high enough to achieve the required efficient wetting.

#### 3.1.2 Wettability of Small Specimens

The tension crack growth specimen is one of the specimens that packed inside the HFTM capsule. It is used in this wettability testing and it has outer dimensions of  $11.5 \times 11.5 \times 4.6$  mm<sup>3</sup>. The objective is to test the wettability of these small specimens when they are placed together as one set. Four different arrangements of the specimens were tested separately in four experimental runs (W1 to W4) as follows and shown in Figure 5. In the first run (W1), four specimens are aligned as one row with inter-distance of 10mm between the specimens. The second run (W2) has six specimens placed as one row again but the inter-distance between the specimens is different (0.4, 0.7, 1.0, 1.7, and 2.0 mm). In the third run (W3), nine specimens are arranged in two alternating orientations (parallel and perpendicular to the facing view). The specimens are very close to each other; therefore the inter-distance is small and ranges from 0.1 to 0.8 mm. The fourth run (W4) has 8 specimens arranged in 4 pairs where each pair features two adjacent specimens. Two pairs are perpendicular to the facing view while the other two are parallel. In all runs, the set up was heated to  $100^{\circ}$ C to liquidize the sodium, and then the specimens were placed. After that heating was continued up to 400°C where the wetting was not completed, and then between 420°C and 430°C, the wetting was completed at all specimens' surfaces and easy to observe. After run W4 was cooled down to 30°C, it was reheated (i.e. run repeating) with most of the specimens already wetted but some side surfaces are not completely wetted.



# 3.1.3 Hysteresis Effect

It was observed that wetting of the parallel plates and rising of sodium in the channel occurs at high temperatures (415°C to 430°C) and then later after the plates and sodium cool down to room

temperature, both wetting of the plates surfaces and sodium rise remain in place without any change (i.e. hysteresis effect). Two examples of this hysteresis effect are shown in Figure 6: (a) parallel plates 3 & 4 and (b) specimens of run W4. Once wetting of the plates and specimens surfaces had occurred there, it remained after cooling; this has been true for all runs. This hysteresis effect is favourable for the wetting of the Eurofer specimens by sodium inside the HFTM capsules because once the specimens are wetted during the filling process at high temperature (430°C); they will remain wetted later on when the temperature decreases. Actually after the experiment, it was difficult to separate the parallel plates from each other at the room temperature as the sodium is holding "glueing" the plates together. Therefore, it was necessary to heat the plates up to 100°C in order to melt the sodium and then separate the plates.



Figure 6: Specimens are still wetted by sodium after cooling to 26°C.

#### **3.2 Sodium Filling**

The experimental setup consists of the following main components: (i) the prototype capsule that is packed with Eurofer specimens, (ii) a stainless steel container for melting the sodium, (iii) three heaters to heat up the setup, and (iv) five thermocouples to measure the temperatures at different locations. In addition, several structural parts are used to support and hold the main components together. The prototype capsule is considered the central part of this setup and it consists of a rectangular steel frame and two sides made of transparent glass as sight windows, see Figure 7. Fused silica glass was selected to serve as a sight window because it has: (i) chemical compatibility with sodium, (ii) working temperature limit up to  $1000^{\circ}$ C, and (iii) high resolution optical clarity for visual observation (e.g. video recording). Two aluminum plates (covers) are used to fix the two glass sides to the frame with the help of several bolts. The frame was manufactured from a plate of Eurofer steel. At the middle area of the steel frame a rectangular volume of  $9.4 \times 40.1 \times 81.5$  mm<sup>3</sup> was milled to house the specimens while another space (expansion volume) with dimensions of  $9.4 \times 40.1 \times 14$  mm<sup>3</sup> is located above the specimens.

These internal volumes and dimensions are similar to those of the original HFTM capsule from the IFMIF/EVEDA design. Also similar Eurofer specimens stack and other parts such as the sodium filling tube are contained in the capsule. The frame has two through holes (7 mm diameter and 185 mm length) to enclose two cartridge heaters (at both sides, see Figure 7) to heat up the capsule to the required temperatures. Each heater (240V and 600 W) has a diameter of 6.3 mm and a length of 177.8 mm. The heater's working temperature is up to 650°C. There are three thermocouples welded around the filling tube and penetrate into the capsule through the specimens stack to measure the temperature at different depths. In addition, a thermocouple is attached to the outer surface of the filling tube. Another thermocouple is used to monitor the sodium container's temperature. The prototype capsule is densely packed with original IFMIF Eurofer specimens including: (i) 24 flat tensile specimens, (ii) 20 tension fracture toughness specimens, (iii) 20 Charpy impact specimens, (iv) 12 round fatigue specimens, and (v) 6 tension crack growth specimens. These Eurofer specimens should have a uniform temperature profile therefore the heat transfer (mainly conduction) should be well understood and predictable. The filling sodium needs to be melted and heated up to the desired temperature so it can flow smoothly inside the capsule. Hence, the solid sodium pieces are brought to a melting container that is connected to the capsule via a relevant valve to control the sodium flow. The sodium melting container consists of: (i) half-nipple with flange, (ii) CF flange with adapter for Swagelok connection, (iii) high-temperature CF copper gasket DN63. In addition, a band heater (63.5 mm  $ID \times 63.5$  mm wide, 240V and 1000W) is placed around the container to melt the sodium.

Filling the capsule with sodium was performed as follows. First, the setup and the sodium were brought into the glove box that filled with dry argon to maintain an oxygen- and moisture-free atmosphere. Second, the required connections of heaters, thermocouples, data acquisition system and computer were implemented. Third, a thermal run (without sodium) was performed to check the performance of all components and relevant measurements. Fourth, the solid sodium bar was cut inside the glove box into small pieces that fit into the sodium container. Fifth, a digital camera was placed outside the glove box for video recording the filling process. Sixth, the two cartridge heaters were turned on to heat up the capsule to 430°C. When the temperature readings from the capsule's three thermocouples approached 400°C, the band heater was turned on to heat up the sodium pieces in the container when the temperatures were as follows: (i) the maximum temperature from the capsule's three thermocouples was

430°C while the minimum one was around 400°C, (ii) the filling tube's temperature was around 140°C, and (iii) the container's temperature was around 130°C. Finally, the sodium started to melt and about four minutes later, it started to flow and fill the capsule. Figure 8 shows the sequence of the sodium filling process versus the time which is set to zero (00 min : 00 sec) at the beginning of sodium appearance at the capsule's bottom. The filling process was very smooth and quite fast, so it took the sodium (01 min : 02 sec) to fill the capsule and penetrate through any gaps. The whole filling process was video recorded and the pictures in Figure 8 were taken from the recorded videos.



Figure 7: The prototype capsule packed with the Eurofer specimens.



Figure 8: The filling sodium flow versus time (min:sec).

After the filling was complete, all heaters were turned off to decrease the capsule's temperature. During the capsule's temperature decreasing (from 380 to 330°C), the sodium's colour changed black. However, with further cooling down, the sodium's colour partially returned back to silver. These sodium colour changes are revealed in Figure 9 where the front and back views of the

capsule are shown after cooling down to room temperature (RT) in the following day. One can see that some areas of sodium are still black while other areas are back to the silver colour. Figure 9 gives also an indication about how well the sodium filled the capsule from the back side. Filling the capsule with liquid sodium has been demonstrated successfully in this experiment.



Figure 9: The capsule's front (F) and back (B) views after cooling down to RT.

# 4. Conclusions

The conclusions to be reached from this experimental study are summarized as follows:

- Sodium was studied experimentally as a new filling material for the capsules of the IFMIF-DONES HFTM,
- An efficient wetting of Eurofer and stainless steel 316L specimens by sodium can be assured when the temperature of the steel-sodium system is 430°C or higher,
- A successful sodium filling of the prototype capsule was demonstrated where the specimens were wetted effectively and the empty volumes were filled with sodium,
- The objective of proof-of-principle testing of capsule filling and operational experience was achieved.

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