

Study of the wettability of fusion relevant steels by the sodium potassium eutectic alloy NaK-78



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ABSTRACT

Several validation activities were dedicated to the High Flux Test Module (HFTM) of the International Fusion Materials Irradiation Facility (IFMIF) at the Karlsruhe Institute of Technology (KIT) in Germany. The HFTM contains 24 capsules packed densely with Eurofer specimens to facilitate their irradiation in the high flux zone directly behind the IFMIF neutron source. The small gaps among the Eurofer specimens are filled by the sodium potassium eutectic alloy NaK-78 to improve the thermal conduction among the specimens and achieve uniform and predictable temperature distribution. As a result of first trials, the filling process of NaK-78 into the specimens' capsule had been identified as an issue worth further investigations. Therefore, the wettability of the steels Eurofer and SS 316 by NaK-78 is experimentally investigated to evaluate the applicability of this concept and identify the favorable conditions. In the experiment, the capillary rise of NaK-78 in a two-parallel-plates channel (gap) is investigated versus the following: (i) temperature of both NaK-78 and the parallel plates from 50°C to 350°C, (ii) machining techniques used for the parallel plates, (iii) thickness of the gap between the plates, and (iv) material of the parallel plates including Eurofer and SS 316. The present experimental results will help in defining the working conditions required to achieve an optimal filling of the IFMIF HFTM capsules with NaK-78 and a complete wetting of the capsules' specimens.

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1. Introduction

The International Fusion Materials Irradiation Facility (IFMIF) is an accelerator-based neutron source that uses lithium-deuterium $\text{Li}(d,xn)$ nuclear reactions to produce a neutron spectrum similar to that expected in the fusion reactor. The mission of IFMIF is to irradiate the fusion materials in irradiation conditions similar to those of the fusion demonstration (DEMO) reactor in order to: (i) provide data of the structural materials needed for the engineering design and construction of DEMO, (ii) contribute to the selection of candidate fusion materials, and (iii) validate the understanding of the irradiation effects on the fusion materials. Next to the IFMIF neutron source in the high flux region, the irradiation of the structural material (e.g. Eurofer steel) specimens will be performed in the High Flux Test Module (HFTM) [1]. The HFTM contains 24 capsules packed densely with Small Specimen Test Technology (SSTT) specimens to facilitate their irradiation. Each

HFTM capsule contains 80 SSTT specimens in a specimen stack that has dimensions of 81 mm × 40 mm × 9.5 mm. A concept was proposed to use the sodium potassium eutectic alloy NaK-78 to fill the small gaps among the SSTT specimens in order to: (i) improve the thermal conduction among all parts and the heat transfer predictability, (ii) achieve uniform and predictable temperature distribution, and (iii) ensure that the thermocouples are in contact with NaK-78 or specimen and not a gap to obtain accurate representative temperatures. As a result of first trials, the filling process of NaK-78 into the specimens' capsule had been identified as an issue worth further investigations. For that reason, the wettability of two structural materials (namely Eurofer and austenitic stainless steel 316) by NaK-78 is experimentally investigated to evaluate the applicability of this concept and identify the favorable conditions. Eurofer belongs to the group of the Reduced Activation Ferritic Martensitic (RAFMs) steels that have swelling resistance and low activation properties; therefore it has been developed to serve as a structural material for the Test Blanket Module (TBM) of the fusion reactor ITER. The austenitic stainless steel 316 is also a candidate structural material for some ITER large components such as the vacuum vessel and the divertor.

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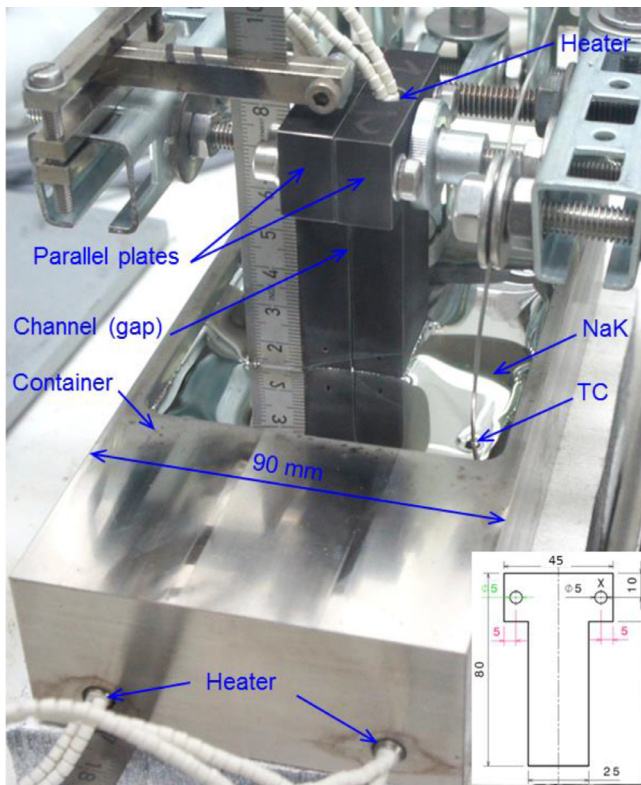


Fig. 1. The experimental setup.

The eutectic alloy NaK-78 (78 wt% K and 22 wt% Na) is in the liquid state at room temperature because its melting point is -12.6°C while its boiling temperature is 785°C under the atmosphere pressure. The density of NaK-78 is 867 kg/m^3 at 20°C and it decreases to 749 kg/m^3 at 550°C [2]. The difficulty of using NaK-78 lies in its volatile reactions with air and water. NaK-78 reacts with oxygen rapidly forming potassium oxide and superoxide which make a crust on its surface. In addition, NaK-78 reacts explosively with water to release hydrogen and form potassium hydroxide and sodium hydroxide while the reaction heat may cause a fire. Therefore NaK-78 should be handled with safety measures and it is usually investigated in a controlled space such as a glove box filled with inert gas such as dry argon or nitrogen. Liquid metals (e.g. NaK-78 and lithium) are main candidates for space reactor systems to serve as a heat transfer medium because of their high thermal conductivity. NaK-78 has good compatibility with stainless steel for temperatures up to 650°C as well as a successful service history for use in space reactor systems [3]. A recent study [4] demonstrated that NaK-78 has also good compatibility with Eurofer steel under IFMIF-relevant conditions such as high temperature of 500°C and continuous service of six months.

2. Experimental setup

The experimental setup, shown in Fig. 1, consists of the following main components: (i) a container made of stainless steel 316L and has a cavity to be filled with NaK-78, (ii) two parallel plates, (iii) four cartridge heaters, (iv) four thermocouples, (v) metallic ruler, and (vi) supporting parts such as bolts, screws, and structural strips. The container has a shape of rectangular prism with outer dimensions of $190\text{ mm} \times 90\text{ mm} \times 40\text{ mm}$ and its cavity has dimensions of $80\text{ mm} \times 80\text{ mm} \times 20\text{ mm}$. The two parallel plates are aligned perpendicularly to the free surface of NaK-78 in the container cavity, see Fig. 1. The geometry and dimensions of

the parallel plates are shown in the inserted drawing of Fig. 1. They have parallel planar surfaces and their thickness is 12 mm which is not shown in the given drawing. The plates are spaced closely and clamped together with a spacer between them using two screws in order to form a parallel-plates channel. The channel (gap) thickness was adjusted to two values of 0.3 mm and 0.6 mm by inserting a steel spacer with dimensions of $30\text{ mm} \times 25\text{ mm}$ and the matching thickness. Due to wettability and capillarity, the liquid NaK-78 may rise in the gap between the immobile parallel plates over its level in the free surface container. The height of the NaK-78 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 temperatures of both the parallel plates and NaK-78 are controlled by implementing four cartridge heaters and four thermocouples attached at different locations within the setup. One short cartridge heater is placed in each plate and two long cartridge heaters are inserted in the container. The long heaters used for the container are high density cartridge heaters with a sheath diameter of 6.4 mm and a sheath length of 178 mm. The heater sheath is made of the standard alloy 321 stainless steel to provide high temperature strength up to 650°C . Ceramic beads insulation, rated to working temperatures up to 650°C , is used for the lead termination. The lead wire is insulated by fiberglass with working temperature of 450°C . The long heaters can provide 600 W with input voltage of 240 V. The short heaters for the parallel plates are high density cartridge heaters with a sheath diameter of 6.4 mm and a sheath length of 76 mm. The short heaters can provide 350 W with input voltage of 240 V. The other specifications are the same as those of the long heaters. The heaters are fed by electricity via four power supplies to control their input voltages. These power supplies are located outside the glove box (where the setup is located) and connected to the heaters by a dedicated feedthrough.

To monitor the temperatures of the setup effectively, four thermocouples were placed in well-chosen and defined locations as follows: (i) one thermocouple is attached to each plate, (ii) one thermocouple is immersed in NaK, and (iii) one thermocouple is in contact with the surface of the container's cavity. The temperatures given in the results are an average of the three thermocouples (two attached to the plates and one immersed in NaK) when reaching a steady state and the thermocouples' readings are in close agreement (i.e. the difference among the readings is less than the thermocouple error). All thermocouples are type K with 1 mm diameter and can withstand temperatures up to 900°C with deviations of $\pm 2.5^{\circ}\text{C}$ up to 333°C and $\pm 0.75\%$ above 333°C as reported by the manufacturer. All experiments were performed inside a dedicated glove box filled with dry argon to maintain an oxygen- and moisture-free atmosphere around the setup which contains NaK-78. The concentrations of oxygen and water vapor, inside the glove box, were monitored and kept as low as $\text{O}_2 < 0.1\text{ ppm}$ and $\text{H}_2\text{O} < 0.1\text{ ppm}$ during all experiments. The glove box is equipped with two feedthroughs needed for the lead wires of the heaters and thermocouples used for the setup inside the glove box.

3. Experiment procedures

The experiment procedures may be summarized as follows:

- The experimental setup and NaK-78 vessel were transported to a dedicated glove box where all experiments were performed because NaK-78 reacts aggressively with air and water. Then the setup's container cavity was filled with NaK-78.
- The parallel plates were immersed in NaK-78 to a depth of 18 mm in a direction perpendicular to the NaK-78 free surface.

Table 1
Characteristics of the parallel plates.

Plate	Material	1st surface machining	2nd surface machining
1	Eurofer	spark erosion, $R_a = 0.30 \mu\text{m}$	spark erosion, $R_a = 4.07 \mu\text{m}$
2	Eurofer	spark erosion, $R_a = 0.32 \mu\text{m}$	spark erosion, $R_a = 3.17 \mu\text{m}$
3	Eurofer	milling, $R_a = 0.63 \mu\text{m}$	grinding, $R_a = 0.70 \mu\text{m}$
4	Eurofer	milling, $R_a = 0.73 \mu\text{m}$	grinding, $R_a = 0.83 \mu\text{m}$
5	SS 316	milling, $R_a = 1.16 \mu\text{m}$	grinding, $R_a = 0.92 \mu\text{m}$
6	SS 316	milling, $R_a = 0.69 \mu\text{m}$	grinding, $R_a = 0.85 \mu\text{m}$

- The heaters, the data acquisition system and relevant software were turned on to record the temperatures of the experimental setup versus time.
- During each experimental run, pictures of the setup were taken at every 50°C (from 50°C to 350°C) after the temperature reached a steady state. Afterwards, the pictures were analyzed and the NaK-78 rise between the parallel plates was determined.
- After each experimental run, the parallel plates were transported outside the glove box for cleaning and removing any traces of NaK-78 using ethanol and an ultrasonic cleaner.

4. Results and discussion

In general, wettability of steels is governed by a number of factors including properties of the spreading liquid, characteristics of the steel surface and conditions of the liquid-solid system such as temperature and surrounding gas. Therefore, the objective of this experimental work is to study the wettability of the parallel plates by NaK-78 via considering the influence of the following factors: (i) temperature of both the parallel plates and NaK-78 within the range of 50°C to 350°C, (ii) machining technique (i.e. spark erosion, grinding, and milling) used to manufacture the parallel plates, (iii) thickness of the gap between the parallel plates, and (iv) material of the parallel plates including Eurofer and SS 316. In this study, the height of the capillary rise of NaK-78 in the gap between the parallel plates is used as a measure of the plates' wettability. In all the experimental runs, the NaK capillary rise and wetting of the parallel plates were observed and pictures were taken versus the temperature at every 50°C starting from 50°C to 350°C. The experimental results of this study are presented in the following sections to show the effect of the above mentioned factors. Table 1 summarizes the parallel plates' characteristics including the plate's material, machining technique used for each surface and the obtained surface roughness parameter R_a (which is equivalent to the average height or depth of the peaks above and below the average centreline of a surface and given in μm).

4.1. Effect of temperature

In the relevant experimental runs, the NaK-78 capillary rise was observed and pictures were recorded versus the temperature increasing from 50°C to 350°C with a step of 50°C. The experimental runs demonstrated that as the temperature of both plates and NaK-78 rises from 50°C to 250°C, NaK-78 does not wet the plates' surfaces and there was no NaK-78 rise in the gap. Between 250°C and 280°C, NaK-78 starts to wet the plates' surfaces and the contact angle begins to decrease (i.e. indicating a start of wetting). At about 300°C, NaK-78 starts to rise in the gap in some experimental runs. The rise of NaK-78 in the gap is obvious and can be measured clearly when the temperature ranges from 300°C to 350°C. It is noticeable that the high temperature improves wetting of the steels by the liquid NaK-78 and the spreading behavior of NaK-78 at high temperatures is completely different from that at room temperature. Table 2 summarizes the results of four experimental

runs dedicated to show the effect of temperature of the parallel plates and NaK-78 on the value of the NaK-78 capillary rise. The increase of the NaK-78 capillary rise with increasing temperature in the experimental runs reflects the obvious effect of temperature. The increase of temperature does influence the properties of NaK-78, for instance its surface tension decreases from 0.15 N/m to 0.12 N/m with the increase of temperature from 20°C to 350°C [5]. Likewise, the viscosity of NaK-78 decreases from 0.94 cP to 0.17 cP with increasing its temperature from 20°C to 550°C [2]. The decrease in the values of both surface tension and viscosity of NaK-78 with the increase of temperature may explain the corresponding improvement in the wettability and hence increasing the capillary rise between the parallel plates. This explanation agrees well with a previous study [6] where it was stated that the main forces which affect a liquid rising in a capillary tube are caused by the gravity as well as the surface tension and viscosity of the liquid.

The present results are in good agreement with the results of some previous research studies [6–9]. For example, in a previous research work [7], the wettability of a solid lithium surface by liquid alkali metals (including sodium, potassium and rubidium) was experimentally investigated. It was observed that the wettability of lithium was improved significantly with increasing the temperature of sodium and potassium. The temperature threshold of wetting was found to be 325°C and 160°C for the lithium-sodium and lithium-potassium systems respectively. Additional article [8] reported that increasing the temperature of several lead-free solders led to an improvement in the wetting of a copper substrate. Similar trend for the wettability dependence on temperature was observed in another study [9], for instance liquid lithium wetted a tungsten surface with a contact angle of 130° at 200°C; however the contact angle was less than 80° at temperatures above 350°C.

4.2. Effect of plates material

The gap between the parallel plates was kept constant at a value of 0.3 mm and the comparison between the two materials (Eurofer and SS 316 steels) of the parallel plates was made at a temperature of 350°C. The machining technique for the compared parallel plates is the same; the first machining is milling while the second machining is grinding. The results, in terms of NaK-78 capillary rise, of these two steels are compared in Table 3. The obtained results show that: (i) with milling, the NaK-78 rise between the Eurofer plates is much smaller than the NaK-78 rise between the SS316 plates (comparing 8 mm with 27 mm) and (ii) with grinding, the NaK-78 rise for both materials is similar (comparing 28 mm to 27 mm). One can conclude that wettability of the Eurofer plates is lower than wettability of the SS 316 plates when the machining technique is milling. In a fusion-relevant previous study [9], experiments were performed to investigate the wettability of SS 316 by liquid lithium. It was found that the wetting temperature (i.e. temperature above which liquid lithium wets the SS 316 surface and below which it does not wet) is 315°C and as the temperature increased beyond this value, the wettability of the SS 316 surface was improved. Unfortunately the literature does not provide any study or data about the wettability of Eurofer steel by NaK-78 or any other liquid metal.

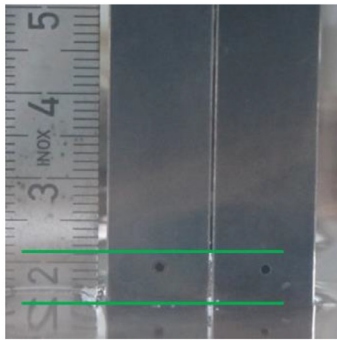
4.3. Effect of machining technique

Influence of the machining technique (milling and grinding) used to manufacture the parallel plates is discussed here in this section and the relevant experimental results are presented in Table 3 where the gap thickness was kept constant (0.3 mm) in all given cases. The present investigation of effect of machining technique on wettability of the steels of interest revealed the following:

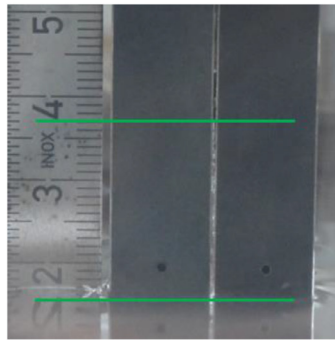
Table 2

Results of the cases dedicated to studying the temperature effect.

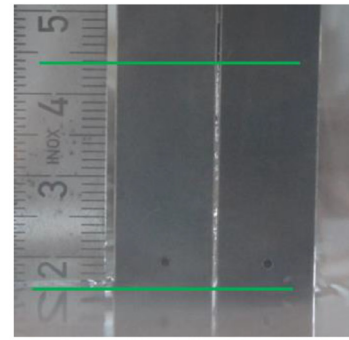
Plates: 3 (grinding) + 4 (grinding), gap: 0.3 mm.



NaK rise = 6 mm at 300°C

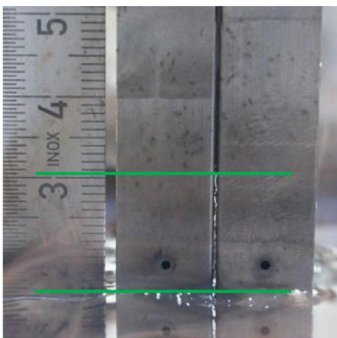


NaK rise = 21 mm at 330°C

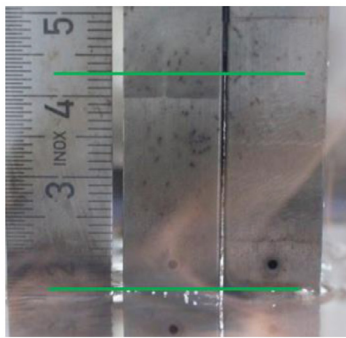


NaK rise = 28 mm at 350°C

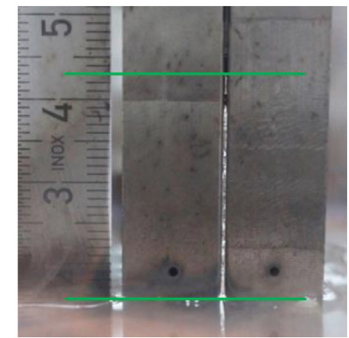
Plates: 5 (milling) + 6 (milling), gap: 0.3 mm.



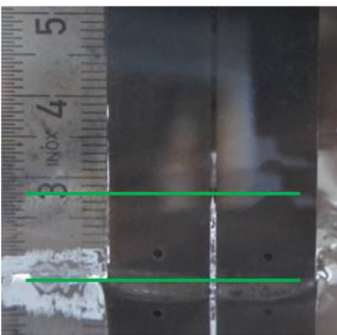
NaK rise = 15 mm at 300°C



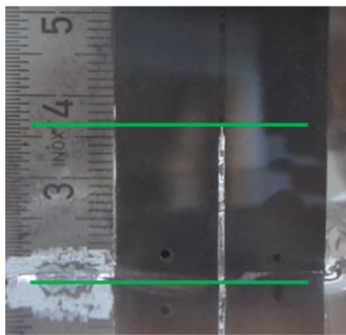
NaK rise = 26 mm at 320°C



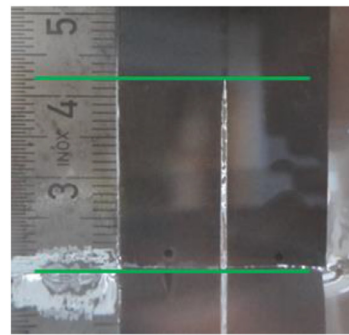
NaK rise = 27 mm at 353°C

Plates: 1 (erosion, $R_a = 0.30$) + 2 (erosion, $R_a = 0.32$), gap: 0.6 mm.

NaK rise = 10 mm at 310°C

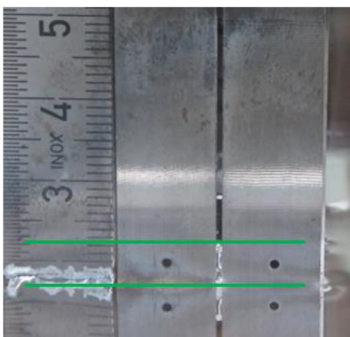


NaK rise = 19 mm at 330°C

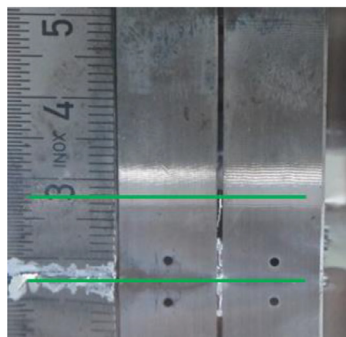


NaK rise = 23 mm at 350°C

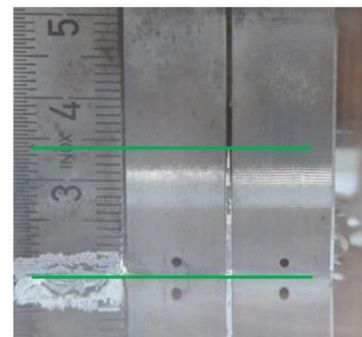
Plates: 5 (grinding) + 6 (grinding), gap: 0.6 mm.



NaK rise = 5 mm at 282°C

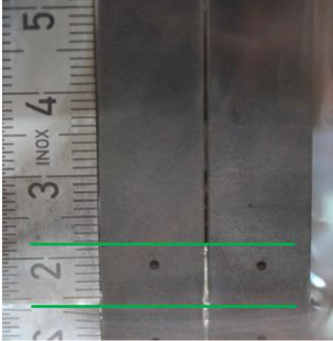
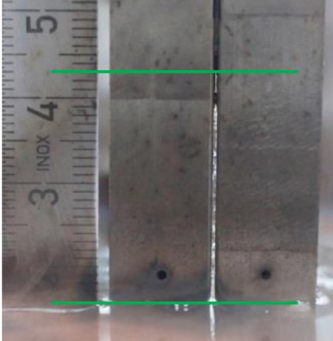
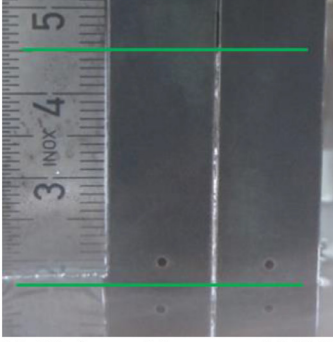
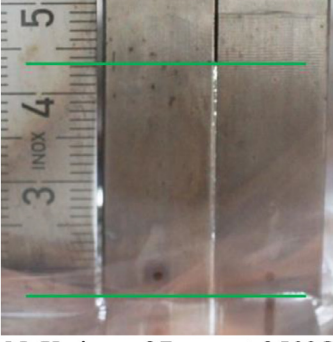


NaK rise = 10 mm at 300°C



NaK rise = 16 mm at 350°C

Table 3
Results that show the effect of plates' material.

Eurofer: 3 (milling) + 4 (milling)	SS 316: 5 (milling) + 6 (milling)
 <p data-bbox="363 629 684 657">NaK rise = 8 mm at 350°C</p>	 <p data-bbox="863 629 1184 657">NaK rise = 27 mm at 353°C</p>
Eurofer: 3 (grinding) + 4 (grinding)	SS 316: 5 (grinding) + 6 (grinding)
 <p data-bbox="363 1078 684 1106">NaK rise = 28 mm at 350°C</p>	 <p data-bbox="863 1078 1184 1106">NaK rise = 27 mm at 350°C</p>

(i) for the Eurofer plates 3 and 4, the NaK-78 rise with the plates machined by milling is much smaller than the NaK-78 rise with the same plates machined by grinding (in specific numbers, it is 8 mm versus 28 mm), and (ii) for the stainless steel 316 plates 5 and 6, the NaK-78 rise is similar with both milling-machined and grinding-machined plates. These results indicate that manufacturing the Eurofer plates or specimens by milling produces a special surface topography (texture) which in turn has a direct impact on the surface wettability. For this reason, a special attention should be given during the manufacturing of the Eurofer specimens for the IFMIF HFTM capsules and the obtained surface texture should be examined carefully.

4.4. Effect of gap thickness

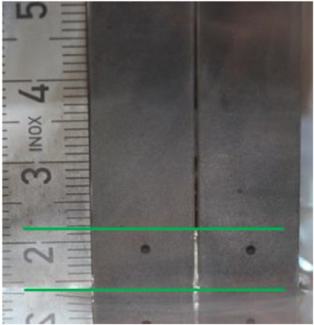
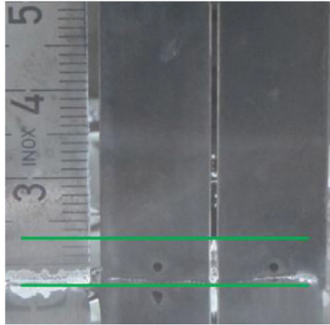
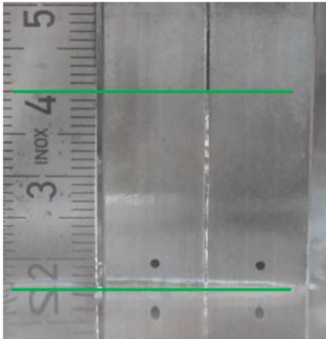
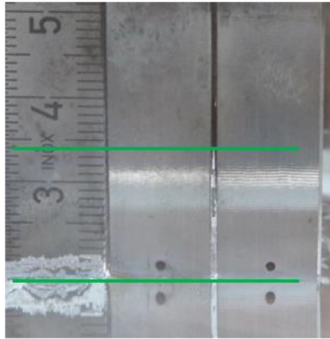
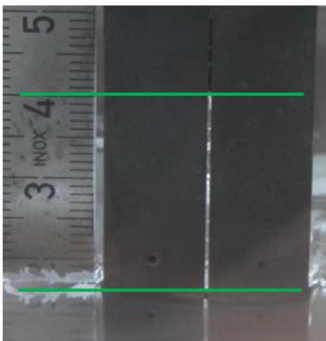
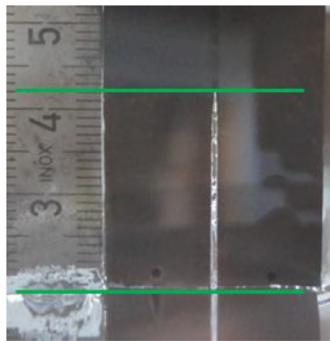
Considering now influence of the thickness of the gap (channel) between the parallel plates, there are three cases: (i) case of Eurofer plates 3 and 4, (ii) case of SS 316 plates 5 and 6 and (iii) case of Eurofer plates 1 and 2. In these cases, all parameters are kept unchanged however each case was investigated with two values (0.3 mm and 0.6 mm) of the gap thickness. The results of these cases are given in Table 4. By comparing the two runs of each case, one can observe that the NaK-78 capillary rise with the 0.3 mm gap is always higher than that with the 0.6 mm gap in all given cases. These results are expected and in agreement with two previous studies dedicated to study the capillary rise between two parallel surfaces. In the first study [10], the effect of the contact angle and capillary size on the equilibrium main meniscus height in rectangular capillaries was investigated. It was demonstrated that the

dimensionless height of the main meniscus decreases with increasing the dimensionless capillary size for the same contact angle. In the second work [11], the capillary rise between two closely spaced parallel surfaces was analysed and it was found that the height of the capillary depends on the spacing between the surfaces and also on the contact angles at both surfaces. Specifically the capillary rise decreased with the increasing the spacing between the parallel surfaces.

4.5. Hysteresis effect

It has been observed that wetting of the parallel plates surfaces and rising of NaK-78 in their gap occurs at high temperatures (frequently between 300°C and 350°C) and then later after both the plates and NaK-78 cool down to room temperature, both the wetting of the plates surfaces and NaK-78 rise remain (i.e. hysteresis effect) without any change. As a demonstration example of this hysteresis effect, Fig. 2 shows the NaK-78 capillary rise and the plates wetting at 350°C as well as at the glove box temperature of around 32°C in the following day. Once wetting of the plates' surfaces had occurred there was no indication that it was vanished with any subsequent cooling, this has been true for all the experimental runs. This hysteresis effect is favourable for the wetting of the Eurofer specimens by NaK-78 inside the IFMIF HFTM capsules because once the specimens are wetted during the filling process at the recommended wetting temperature (350°C); they will remain wetted later on when the capsule is cooled down after finishing the filling process.

Table 4
Results obtained for the effect of gap thickness.

Gap = 0.3 mm	Gap = 0.6 mm
3 (milling) + 4 (milling)  NaK rise = 8 mm at 350°C	3 (milling) + 4 (milling)  NaK rise = 5 mm at 350°C
5 (grinding) + 6 (grinding)  NaK rise = 24 mm at 350°C	5 (grinding) + 6 (grinding)  NaK rise = 16 mm at 350°C
1 (erosion 0.30) + 2 (erosion 0.32)  NaK rise = 24 mm at 350°C	1 (erosion 0.30) + 2 (erosion 0.32)  NaK rise = 23 mm at 350°C

4.6. Wettability and contact angle

The contact angle of liquid on a solid surface is used as a measuring parameter to describe how well (i.e. degree of wetting or wettability) the liquid spreads over the surface. It is defined as the angle made by the intersection of the liquid-solid interface line and the liquid-gas interface line. When the liquid spreads well on the surface, the contact angle is small while a large contact angle is observed if the liquid beads on the surface. For instance, a contact angle less than 90° implies that the surface wetting is favourable, and the liquid spreads well on the surface. On the other hand contact angle larger than 90° indicates that the surface wet-

ting is unfavourable and the liquid decreases its contact with the surface forming a compact droplet. Table 5 shows the change of contact angle versus the temperatures (i.e. at 50°C and high temperatures from 250°C to 350°C) for three different cases from this study. As to be expected from literature data and previous experiences, at low temperatures NaK-78 has a high surface tension which makes large amounts of it tend to ball up on the surface; hence it does not wet the surface well. This can be clearly seen in the pictures of the left column of Table 5, where NaK-78 forms a convex meniscus (capillary depression) at the outside surface of the steel plate and the contact angle is bigger than 90° at 50°C . On the other side (right column of Table 5) at high temperatures from

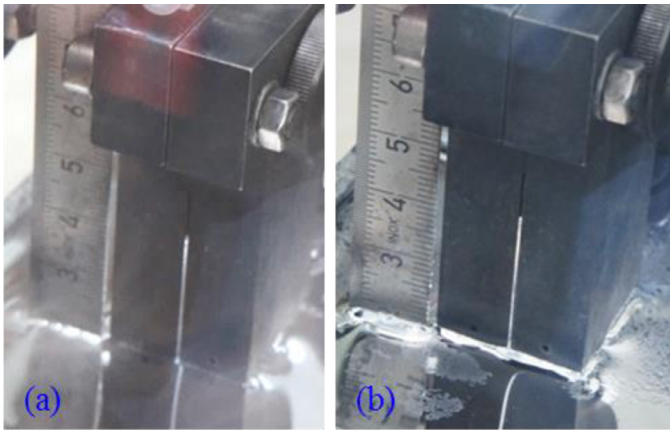


Fig. 2. NaK capillary rise: (a) at 350°C and (b) at 32°C in the following day for plates 1 (erosion 4.07)+2 (erosion 3.17).





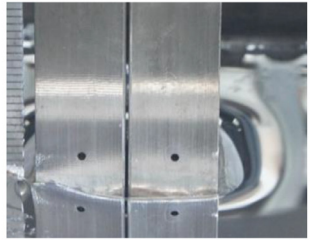

250°C to 350°C, the contact angle is less than 90° and the meniscus straightens out where the surfaces are wetted clearly. All other experimental runs demonstrated similar behaviour of the contact angle change during increasing the temperature of the steel-NaK-

78 system. In the present experimental setup, NaK-78 elevates in the gap between the two parallel plates when both contact angles (i.e. contact angle between NaK-78 and each plate) are less than 90°.

4.7. Surface wetting

In the previous sections, the experimental results were given in form of pictures showing the capillary rise of NaK-78 between the parallel plates but the wetting status of the internal (facing) surfaces was not given. Therefore, wetting of the internal surfaces of the parallel plates, in some experimental runs as an examples, is given in this section and presented in Fig. 3. These pictures give clear views of the wetted surfaces as an evidence of a complete surface wetting (i.e. NaK-78 spreads well on the plates' surfaces covering the whole plate width of 25 mm). Similar results of a complete wetting of the plates' internal surfaces were obtained in all experimental runs. These results are important for the NaK-78 filling of the IFMIF HFTM capsules because it has been experimentally proved that a complete wetting of the capsules specimens by NaK-78 can be achieved.

Table 5
Change of contact angle versus temperature.

Plates: 1 (erosion 0.30) + 2 (erosion 0.32), gap = 0.3 mm	
	
contact angle > 90° at 50°C	contact angle < 90° at 300°C
Plates: 3 (milling) + 4 (milling), gap: 0.6 mm	
	
contact angle > 90° at 50°C	contact angle < 90° at 250°C
Plates: 5 (grinding) + 6 (grinding), gap = 0.3 mm	
	
contact angle > 90° at 50°C	contact angle < 90° at 350°C

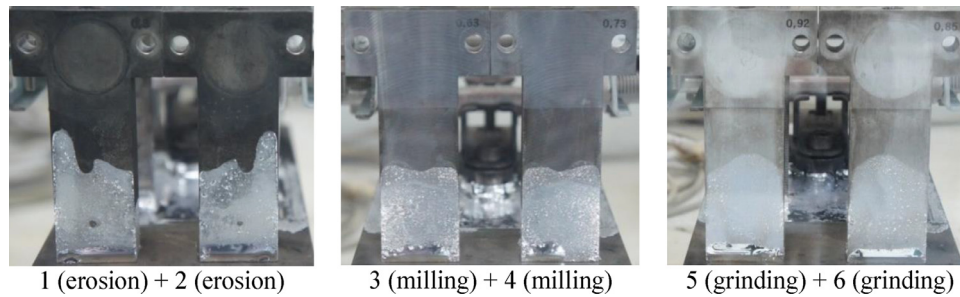


Fig. 3. Complete wetting of the surfaces of the six plates.

5. Conclusions

The following conclusions can be drawn from the present study:

- Wettability of fusion relevant steels (Eurofer and SS 316) by the sodium potassium alloy NaK-78 was experimentally studied within the temperature range of 50°C to 350°C.
- The wettability of the steels' surfaces is governed by some parameters; however it is mainly affected by the temperature of the steel-NaK-78 system.
- Wettability of Eurofer by NaK-78 under IFMIF-relevant machining and surface roughness can be assured when the temperature of the Eurofer-NaK-78 system is 350°C or higher. Wetting was also observed at lower temperature (e.g. 280°C) in some cases.
- The present results will help in defining the working conditions required to achieve an optimal filling of the IFMIF HFTM capsules with NaK-78 and a complete wetting of the capsules' specimens.

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