



Ricerca di Sistema elettrico

## Erosion/corrosion of SIC and SIC/SIC in liquid metal

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## EROSION/CORROSION OF SiC AND SiC/SiC IN LIQUID METAL - ANNUAL REPORT 2012

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Accordo di Programma Ministero dello Sviluppo Economico - ENEA

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Area: Produzione di energia elettrica e protezione dell'ambiente

Progetto: Attività di fisica della fusione complementari a ITER

Obiettivo: Progettazione e costruzione di una camera sperimentale per prove di erosione-corrosione ad alta temperatura (1000°C) di campioni di SiC/SiC in litio-piombo

Responsabile del Progetto: Aldo Pizzuto, ENEA

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## Sommario

This report describes the activities performed during the period Oct. 2012 – Sept. 2013 in the framework of the task “Erosion-corrosion of SiC and SiC<sub>f</sub>/SiC in liquid metal” of the DEMO R&D on SiC/SiC Composites for the IFERC Project.

Based on the results of a literature survey, the experimental plan and the technical requirements of the experimental apparatus have been defined also considering the recommendation of the “Peer Review”.

Main characteristics of the design of the apparatus and the updated status of its construction are reported in this document. Particularly, the thermal map of the oven studied through a finite element code permitted the design of the heating system to be completed. The preliminary control scheme of the apparatus has been modified and its new version is described and commented in this report.

The construction of the oven has been completed and the oven has been delivered at ENEA Frascati laboratories where preliminary tests (control system, sweeping gas and vacuum system, heating systems) have been carried out. During these tests, a failure of the heater resistance occurred. The repair will consist in the substitution of the entire sub-assembly composed of the resistance plus terminals plus alumina block with a new one fabricated by the US supplier. As a consequence, the acceptance tests to be carried out in Frascati on mid October have been postponed by three months.

## 1. INTRODUCTION

SiC fiber reinforced SiC matrix composites (SiC<sub>f</sub>/SiC) are candidate materials for applications in fusion power reactor because of their favorable thermal-mechanical and low activation properties [1]. Especially, these composites exhibit interesting characteristics as both functional and structural materials in the designs of DEMO blankets and of test blanket modules of ITER. The key aspects to be studied for these applications concern the fabrication technology, irradiation effects, mechanical characterization and the study of chemical/physical interactions with the operating environments. Accordingly, the Broader Approach (BA) program is considering several R&D activities on SiC materials [2].

Among the other applications, SiC composites are proposed both as flow channel inserts of liquid blankets to reduce corrosion and the MHD pressure drop and as a high temperature (1000–1100 °C) structural materials [3-5].

In the framework of the DEMO R&D on SiC/SiC Composites for the International Fusion Energy Research Centre (IFERC) Project, a study of the erosion-corrosion of SiC materials in Li-Pb has been planned. This study foresees the construction of an experimental apparatus for performing high temperature tests of rotating SiC materials into the liquid metal. The experimental apparatus will be designed and built at ENEA Frascati laboratories while the tests will be carried out at the Rokkasho IFERC site.

## 2. RECOMMANDATION FROM “Peer Review”

Concerning the operating parameters of the oven, the Final Report of the IFERC DEMO R&D Peer Review (Sept. 2012) recommended the following actions to be taken: (i) to keep the test temperature below the value of design-temperature plus some 50/100 K (i.e. below 750–800 °C); (ii) to investigate the effects of surface conditions (fibres penetration, oxidation etc), coating on fibres and impurities (e.g. trace elements that exist in irradiated environment and FM (Ferritic-martensitic) steel-corrosion products).

Accordingly to the first comment, the operating temperature of the tests will be 800 °C: such an operating condition does not affect the present design that foresees 1000 °C as maximum temperature.

The second comment will concern the analysis of the samples and the Li-Pb path to be carried out during the experiment in the Rokkasho laboratories.

### 3. DESIGN OF THE EXPERIMENTAL APPARATUS

The design of the oven has taken into account the main characteristics of the materials used and their interactions at high temperature. Over 1000 °C, neither metal nor ceramic crucible different from SiC can be used because of the formation of undesired compounds with the liquid metal: for instance, Mo in presence of the SiC sample and LiPb could form molybdenum carbides [6]. Based on these considerations, both the crucible containing the LiPb bath and the lower part of the rotating bar holding the specimen are made of high density monolithic SiC, see fig. 3-1. The rotating bar is made of stainless steel in its upper part (before entering the crucible), while it consists of monolithic SiC in its lower part which enters the SiC crucible where the contact of the LiPb vapor could occur. The specimen is a disk of the SiC composite to be tested: such a configuration of the specimen rotating into LiPb permits to minimize the dragging of the liquid and then maximize the relative velocity between the SiC specimen and the liquid metal. Particularly, the peripheral velocity of the disc has been assumed as the relative velocity between the SiC specimen and the liquid LiPb.

The operation under vacuum could be favorable for a better thermal insulation of the chamber; however, it cannot be applied because of the high vapor pressure of the lithium alloy. Accordingly, the experiments are carried out under inert gas atmosphere (Ar) while vacuum pumping of the oven is foreseen for sweeping the air before starting the test.

The high temperature oven is a water-cooled stainless steel chamber in which the SiC crucible containing the liquid metal is surrounded by heating elements.

A steady state calculation based on a difference-finite analysis has been carried out in order to evaluate the temperature profile inside the oven. The oven has been divided into a number of sections consisting of nodes where the energy balances have been performed. These energy balances have been assessed by an algebraic equation describing the temperatures of each node in terms of the neighboring nodal temperatures, the geometry, and the thermal properties. Particularly, the equations have been approximated by the finite-difference technique in which  $n$  algebraic equations for the  $n$  nodes have been solved by an iteration method.

Fig. 3-2 shows the scheme of the experimental setup and the thermal map obtained through the finite-difference analysis in which the temperature of the heating elements has been fixed at 1400 °C, the external temperature (air) at 30 °C. The heat transfer coefficient air/stainless steel has been assumed to be  $30 \text{ W m}^{-2} \text{ K}^{-1}$  while no cooling of the chamber has been considered (conservative hypothesis). Under these conditions, the temperature of the lower part of the crucible where the SiC specimen rotates into the liquid metal is around 1000 °C that is the design temperature (higher than the operating temperature which is 800 °C).

Especially, the heating elements consist of a dense cermet material made of  $\text{MoSi}_2$  (molybdenum disilicide) and an oxide glassy phase component. Such materials are very stable and efficient: in fact, they can operate up to 1600 °C in air and provide rapid temperature ramping. Inside the chamber the thermal insulation is obtained through polycrystalline alumina shields. An external brushless motor permits the rotation of the SiC specimen through the support bar described above. The specimen has to be sunk into the LiPb at high temperature and extracted before cooling when the metal is liquid: accordingly, the rotating bar with the specimen is moved up-down through an external sliding system connected to the oven via an edge welded bellow feedthrough.

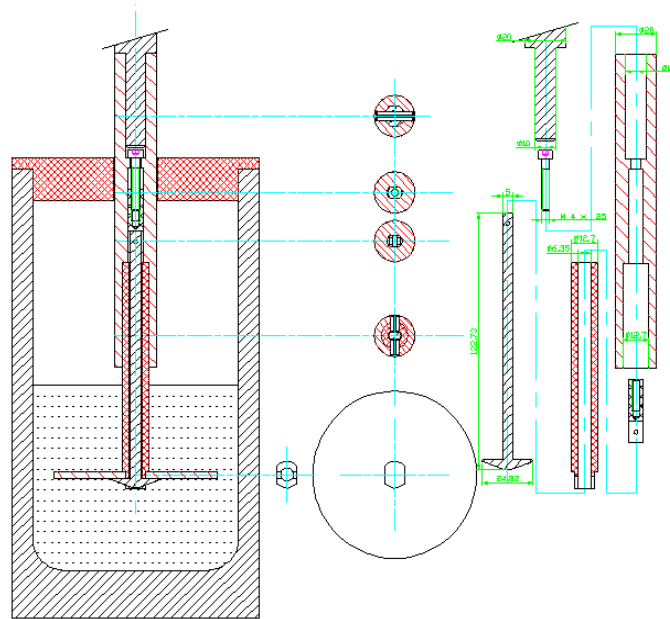


Fig. 3-1 - Draft of the design of the crucible, the SiC specimen and the rotating bar.

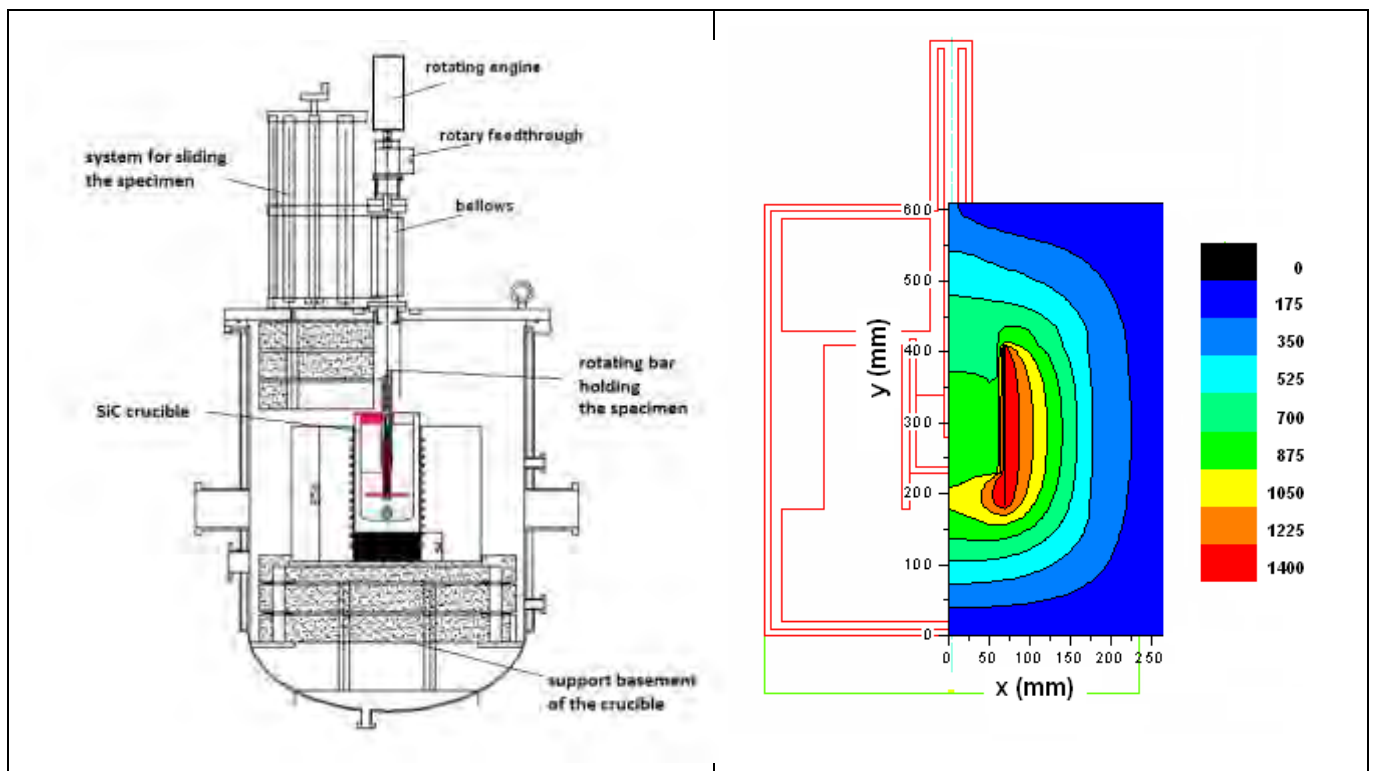


Fig. 3-2 - Draft of the oven (left) and its thermal map (right).

### 3.1 Main characteristics and interfaces of the experimental device

By resuming, the main characteristics of the oven are:

- dimension of oven and support frame H 0.90 x L 0.90 x W 1.50 m, dimension of power supply and control rack H 1.60 x L 0.50 x W 0.80 m,
- the maximum power consumption for the equipment is about 16 kW 10 kW for the heater and 6 kW for the motor,
- about 3 m<sup>3</sup> (STP) of inert gas (Ar) will be required at start of the experiment in order to ensure controlled atmosphere conditions (procedure of vacuum pumping and filling the oven with inert gas) and, after the start-up, about 0.5 L (STP)/min of Ar will be required for normal operation,
- about 3 L/min of water at ambient temperature will be required for cooling the oven chamber.

#### 3.1.1 Electrical connections

The electrical connections proposed by the JA Implementing Agency (IA) and described in the file "Electrical connections\_120130.pptx" (sent by email on Jan 31) are accepted by ENEA. These electrical connections make available 25.7 kVA (20.6-23.1 kW for phase factor of 0.8-0.9) of power for the motor and 17 kW of power for the heater.

#### 3.1.2 Electrical scheme provided by EU(ENEA)

Accordingly to the scheme of the electrical connections proposed by the JA-IA described in the previous section 4.1.1., the general electrical scheme of the experimental apparatus will consist of:

- heating power 10 kW,
- motor power 6 kW.

Such an electrical scheme fulfills the connections proposed by the Japanese Part.

#### 3.1.3 Control scheme

The control scheme of the apparatus is shown in Fig. 3.1.3-1. The control system is operated through a National Instruments control board connected to a personal computer (PC). The control of the temperature is carried out independently by a T controller in order to ensure its availability also in case of the PC default. Other main controls concern the Ar gas and cooling water flow rates.



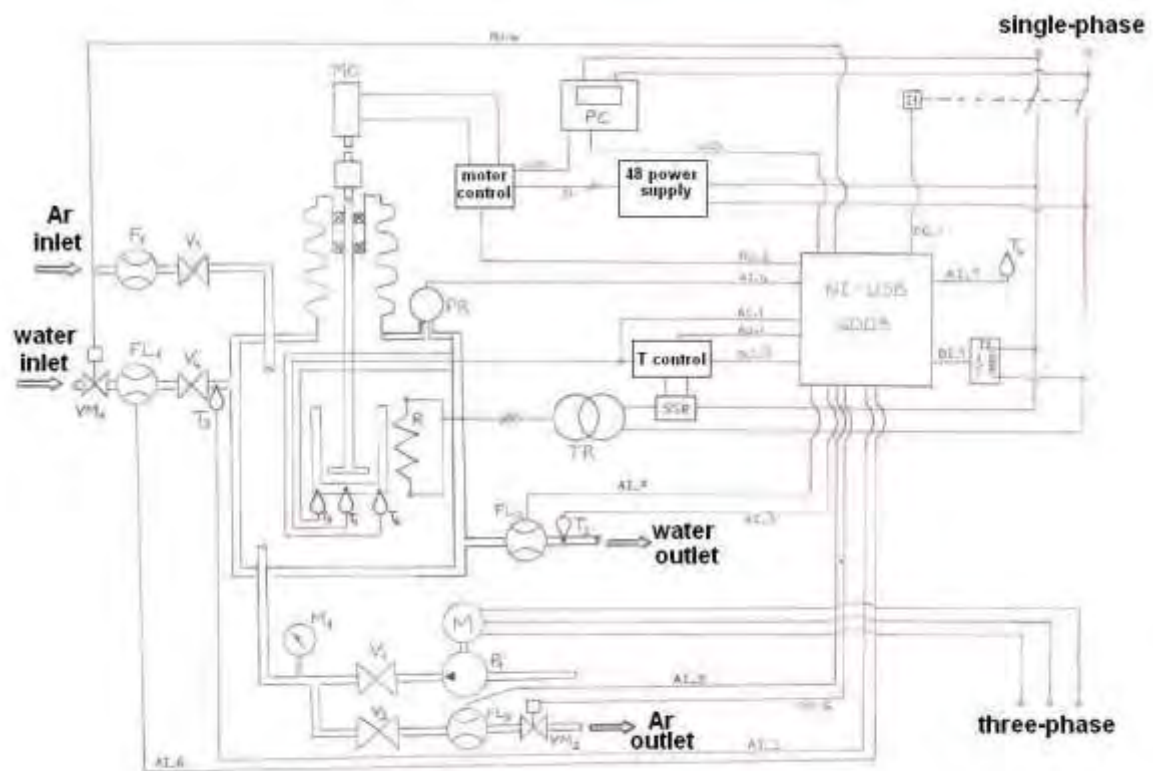


Fig. 3.1.3-1 – Control scheme of the apparatus.

List of the symbols of Fig. 4.1.3-1

F1, F2= gas flow meter

V1, V2, V3, V4= valve

VM1, VM2=electric valves

M1=manometer

FL1, FL2, FL3=gas/liquid flow controller

P1=vacuum pump

PR= pressure gauge

R= heating coil (6 kW 90V)

TR= electrical transformer 220V 50Hz 90V 50Hz

SSR= solid-state relay

MO= motor for rotating the specimen

I1, I2= electric interruptor

T1-T5= thermocouple

NI-USB 6009= control board (National Instruments)

AI.n= analog input

AO.n= analog output

DI.n= digital input

DO.n= digital output



#### 4. STATUS OF THE EQUIPMENT CONSTRUCTION

The construction of the experimental apparatus has been completed and it has been delivered and installed at ENEA Frascati laboratories (see fig. 4-1 and 4-2) where preliminary tests have been carried out.



Fig. 4-1 – Delivery at ENEA Frascati of the apparatus.



Fig. 4-2 – Installation at ENEA Frascati laboratories of the apparatus.



Fig. 5-3 – Experimental chamber: detail of the flange with the sliding and rotating system.

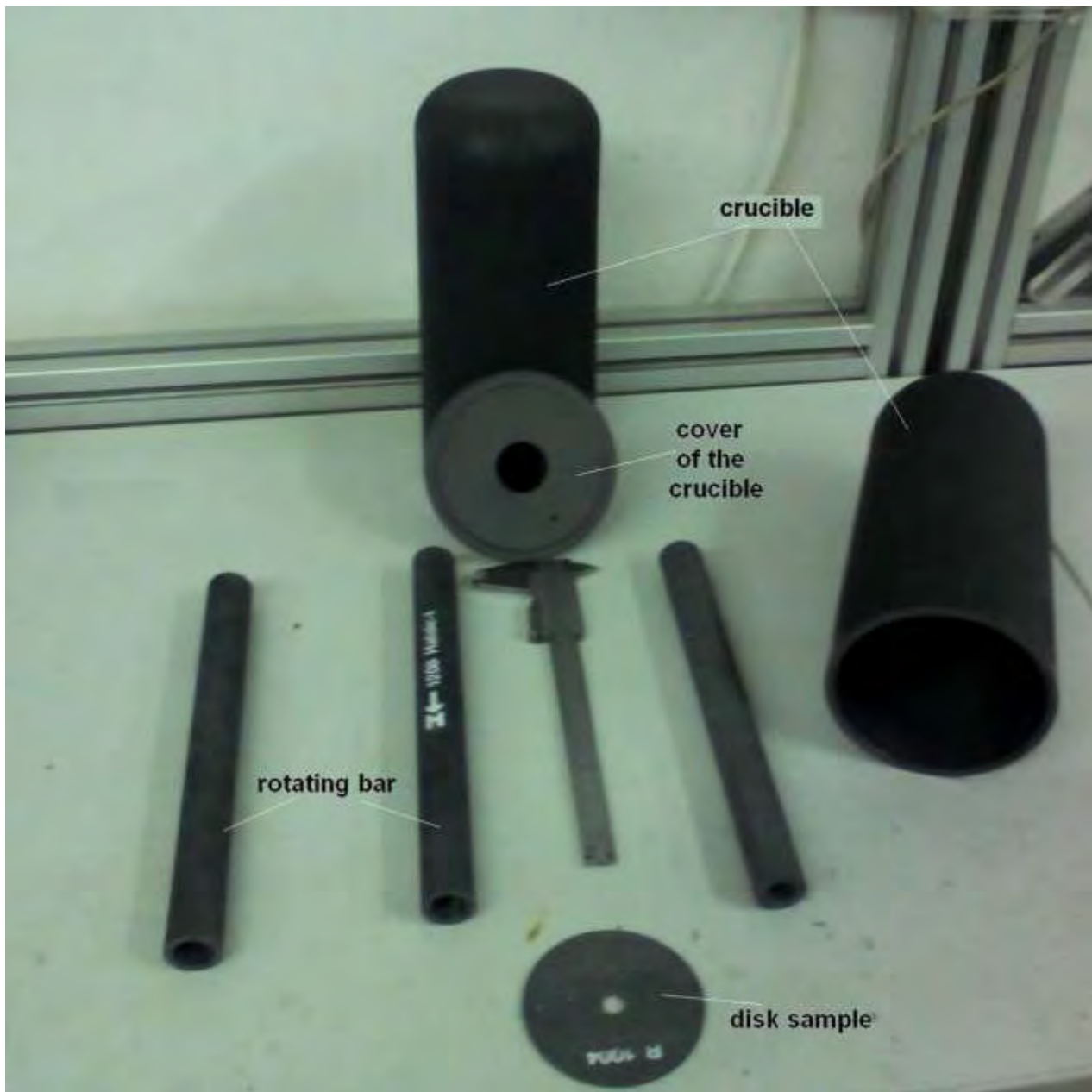


Fig. 5-4 – View of the crucibles, rotating bars (lower part in monolithic SiC) and a disk sample.

Preliminary testing at ENEA laboratories concerns mainly the control and heating systems. The assembly and the rotation of the SiC crucible and sample disk have been also verified. Fig. 5-3 shows the details of the sliding and rotating system while fig. 5-4 depicts the crucible, rotating bar and sample disk.

During the preliminary tests carried out in presence of the Japanese colleagues, a failure of the heater resistance occurred. In order to repair the apparatus, the substitution of the entire heating sub-assembly is needed. The US supplier of the heating sub-assembly (the resistance plus terminals plus alumina block) required at least two months for delivering a new device at Frascati laboratories. Accordingly, ENEA is requiring to F4E and JAEA to postpone on end of January 2014 the acceptance tests previously planned to be carried out in Frascati on mid October.

## REFERENCES

- [1] B. Riccardi et al., Issues and advances in SiCf/SiC composites development for fusion reactors. *Journal of Nuclear Materials* 329-333(2004) 56-65.
- [2] T. Nishitani et al., Recent progress in blanket materials development in the Broader Approach activities, *Journal of Nuclear Materials* 417 (2011) 1331–1335.
- [3] R. Naslain, “Materials design and processing of high temperature ceramic matrix composites: State of the art and future trends”, *Adv. Compos. Mater.* 8/1 (1999) 3-16.
- [4] J.-F. Salavy et al., The HCLL Test Blanket Module system: Present reference design, system integration in ITER and R&D needs, *Fusion Eng. Des.* 83 (2008) 1157–1162.
- [5] S. Nishio et al., Improved tokamak concept focusing on easy maintenance, *Fusion Eng. Des.* 41 (1998) 357–364.
- [6] B.A. Pint, J.L. Moser, P.F. Tortorelli, Investigation of Pb–Li compatibility issues for the dual coolant blanket concept, *Journal of Nuclear Materials* 367–370 (2007) 1150–1154.