EQUINOX

A novel process for manufacturing complex shaped Fe-Al intermetallic parts resistant to extreme environments

Volume 2

Horizon 2020
European Union funding
for Research & Innovation

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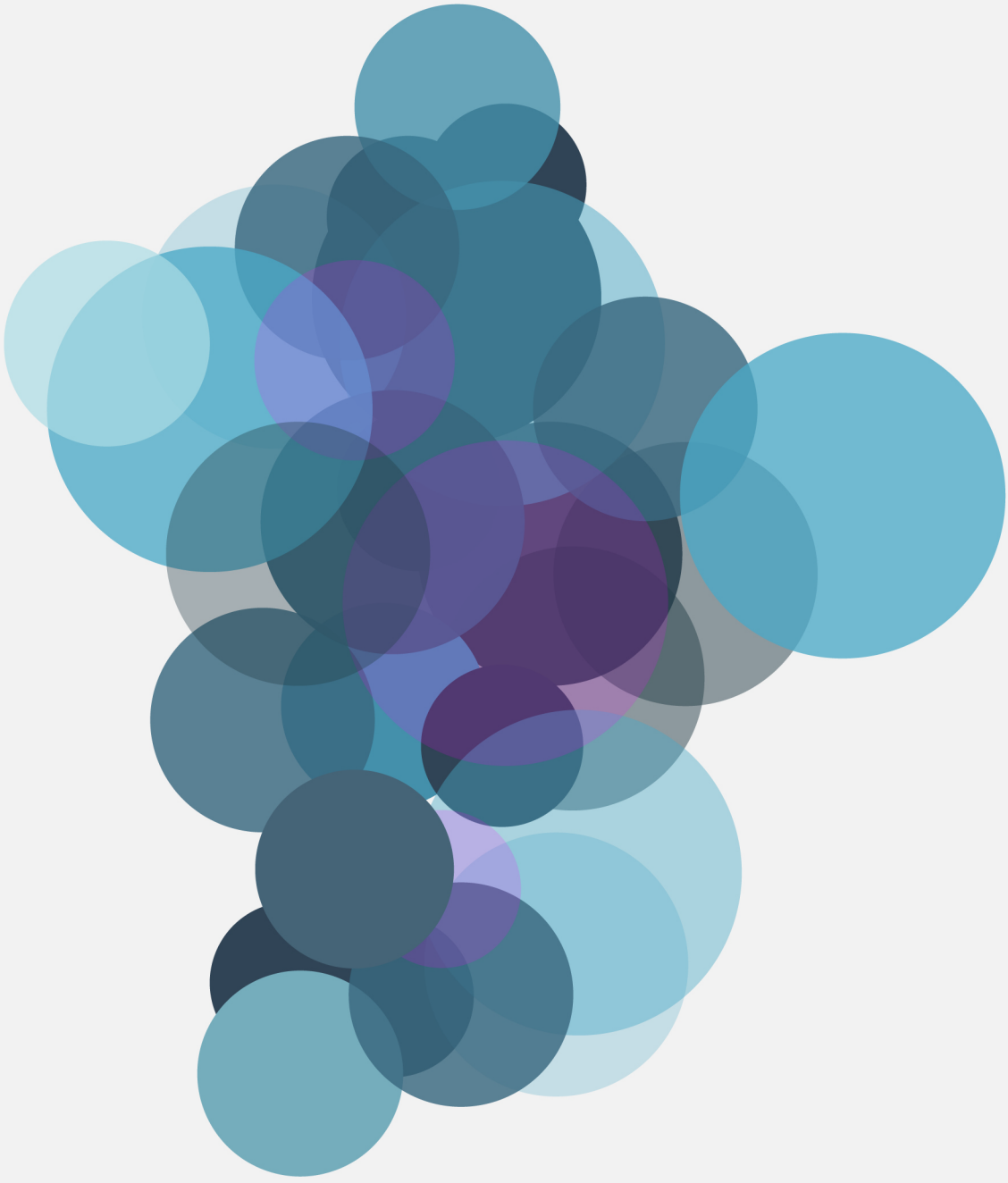

A novel process for manufacturing complex shaped Fe-Al intermetallic parts resistant to extreme environments

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Project background

After World War II some east European countries were facing severe problems to buy Cr and shortage Ni on the free market to cover their needs for stainless steel production and materials for high temperature using.

To overcome this shortage countries of Eastern Block initiated a research in national research institutions, universities and local industry in the 50s – 60s to develop low cost alternatives for heat resistant cast iron and stainless steel alloys based on intermetallics casted from accessible and cheap Iron, Aluminium and Carbon.

These efforts result in the past in the materials such as Thermagal© Tchugal© and Pyroferal©. Pyroferal© offered quite impressive results on high temperature corrosion resistance. It was tested against various severe conditions, such as air atmosphere, vanadium pentoxide, molten glass, carburization, nitration and the atmosphere of the natural gas cracking generators.

Though Pyroferal© was manufactured only by casting, welding was the important procedure not only to produce complicated shapes, but also to repair the faults in casts. Unfortunately, the practical use of these materials was limited due to various problems.

These problems related to instability and welding could not be overcome by state of the art in material science at that time.
In the 60s, access to Chromium was no problem any longer and the dust of history covered the knowhow on (pre-) industrial use of FeAl. But things may change again. Chromium and Nickel are listed in the table of
CRMs with a current projected lifetime of 25 - 100 years.

Project overview

EQUINOX tries to blow away the dust of history from this early work on FeAl, aiming to combine latest state of the art in intermetallic metallurgy to overcome the problems that our ancestors were facing when they failed to translate unique corrosion and wear properties of FeAl into a low cost Cr/Ni-free alternative for stainless steel products.

There is a need to find solutions to replace Critical Raw Materials (CRMs) such as Chromium, Nickel, Molybdenium and Vanadium in high volume end consumer products. Steels and superalloys with considerable amounts of these CRMs are widely used in many industrial applications, particularly under extreme conditions where corrosion and wear resistance are needed.

It is generally accepted, that intermetallics in particular low cost FeAl offer outstanding material properties. Unfortunately it is difficult to translate their properties to real products, as intermetallics suffer from low ductility at ambient temperature and poor machinability.

The impact of FeAl intermetallics as a low cost Cr-free alternative for stainless steel would therefore be much higher if a cost effective industrial process would beavailable, that allows to manufacture complex 3-D geometries of almost unlimited shapes from small grain size (0.1-5 µm) high ductility material.

Objectives

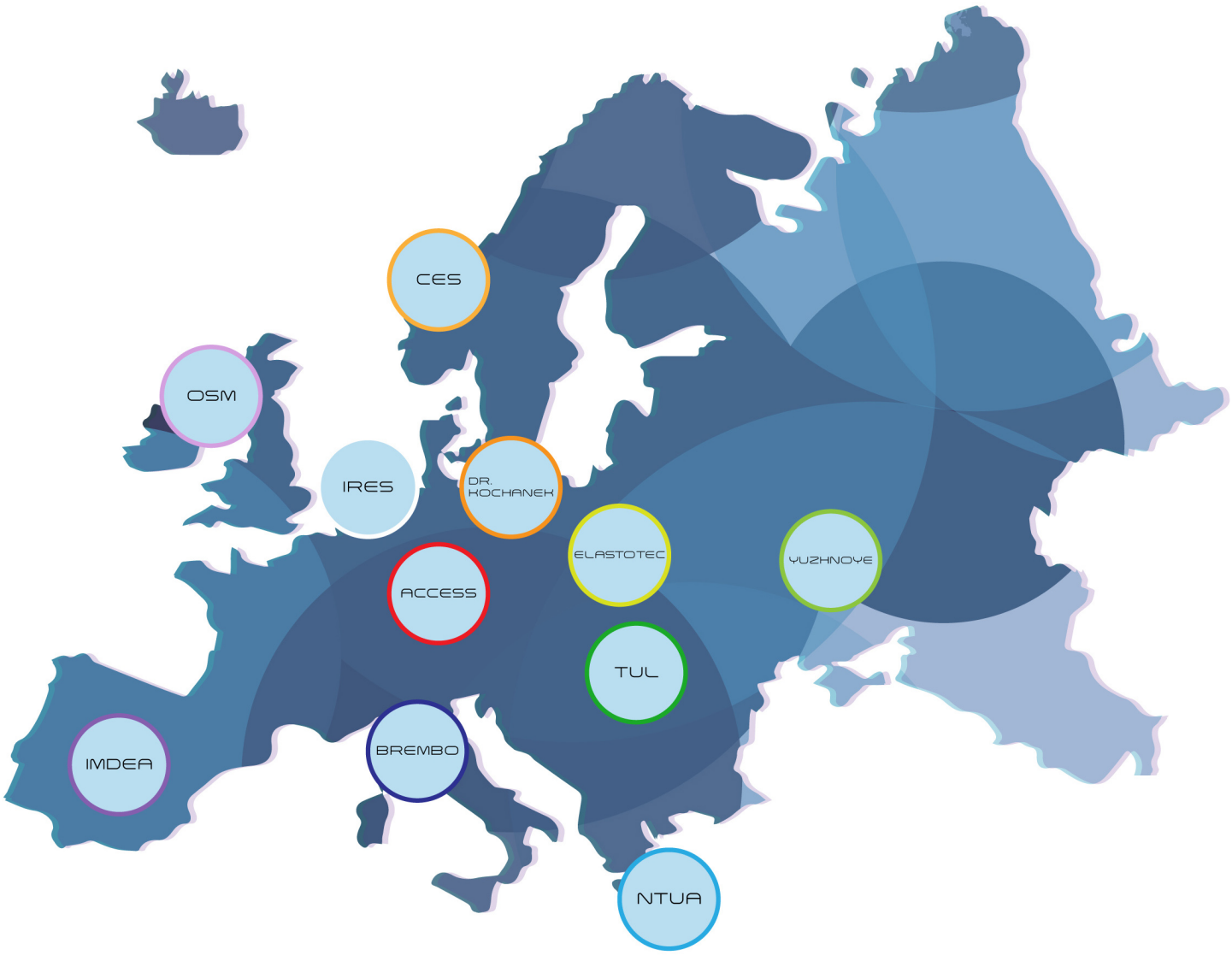
The main objective of EQUINOX is to develop a novel process that allows to substitute Cr/Ni based (stainless) steel parts used in high volume end consumer products such as in the lock industry, electronics, process industry and automotive industry with a novel near net shape production technology for a new class of highlyadvanced ductile Fe-Al based intermetallics.

To produce extremely fine grained FeAl-Material with high ductility via reactive infiltration of porous iron preforms with liquid Aluminium.
To understand how ultrafine particle based porous iron structures of complex 3D-shape may be tailored to be used as optimized preforms for reactive infiltration of liquid Al-alloys.
To develop a reactive infiltration process by using two different techniques: suction and centrifugal casting.
To simulate reactive infiltration process by physically based multi-scale models based on StarCast and MICRESS.
To optimize mechanical properties of **EQUINOX** material with respect to microstructure based on process conditions and consecutive heat treatment.
To scale up the process from lab to small pilot plant with respect to the industrial needs.
To transfer the concept to at least one real demonstrator which will be tested for high corrosion and wear resistance.
To evaluate the industrial impact of **EQUINOX-concept** with respect to economic as well as technical aspects.l.

Concept and approach

The EQUINOX process well fits into "Growing a Low Carbon, Resource Efficient Economy (1) with a Sustainable Supply of Raw Materials (2) - for materials under severe conditions (3)" by many aspects:

No CO2 is produced as side product as oxygen from raw material iron-oxide is fixed as H2O.
No waste of material: 100 % of the material that enters into the process chain ends up as final product of complex 3 D-shape.
No energy is used for "material tourism" All steps of the process run at one single location – Fe3O4 and Al entering through the front door and final 3D-shaped Intermetallic parts leaving through the back door.
Solar heat may be used to cover most part of energy input as temperature level is just slightly above the melting point of Al in all steps involved.
The process may be completely based on H2(which could be made from renewable energy).
Materials with properties close to stainless steel are manufactured from abundant Fe and Al - without (or at least drastically reduced) CRM-materials Cr/Ni/Mo.
EQUINOX materials exhibit properties that withstand corrosion, cavitation and wear offering good results on LCA (life cycle analysis).



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Project Partnership

Eleven partners participate in the EQUINOX project, representing academic, applied research and industrial development.

NATIONAL TECHNICAL UNIVERSITY OF ATHENS,
Greece

ELASTOTEC GmbH ELASTOMERTECHNIKEN,
Germany

DR. KOCHANEK ENTWICKLUNGSGESELLSCHAFT,
Germany

FUNDACION IMDEA MATERIALES,
Spain

TECHNICKA UNIVERZITA V LIBERCI,
Czech Republic

ACCESS e.V.
Germany

OPEN SOURCE MANAGEMENT Limited,
United Kingdom

CES OPERATIONS,
Norway

FRENI BREMBO Spa,
Italy

YUZHNOYE DESIGN OFFICE NAMED AFTER MIKHAIL YANGEL,
Ukraine

IRES,
Belgium.

Running the project

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ACCESS e.V.

Using computational thermodynamics, microstructure simulations and computational fluid dynamics as modelling and simulation tools we obtained deeper understanding of the kinetics of solidification, the formation of intermetallic phases and the dynamics of melt infiltration into the porous preforms.

Parameters derived from these theoretical studies led to successful infiltration of iron-aluminum melts into porous iron preforms.Pictures from the centrifugal casting experiments (shown right) indicate excellent infiltration results. The next steps will deal with improving the efficiency of the infiltration and reaction control leading to the desired intermetallic phases

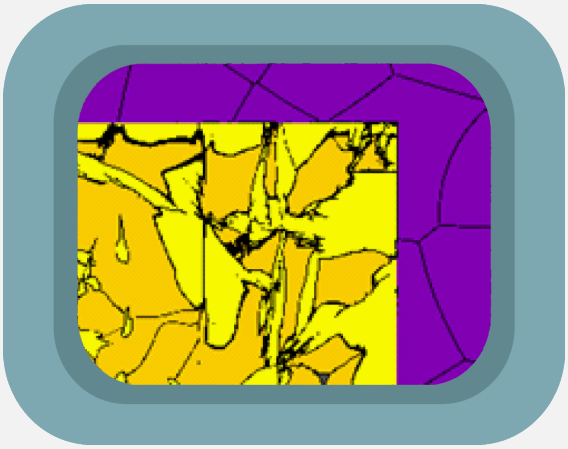
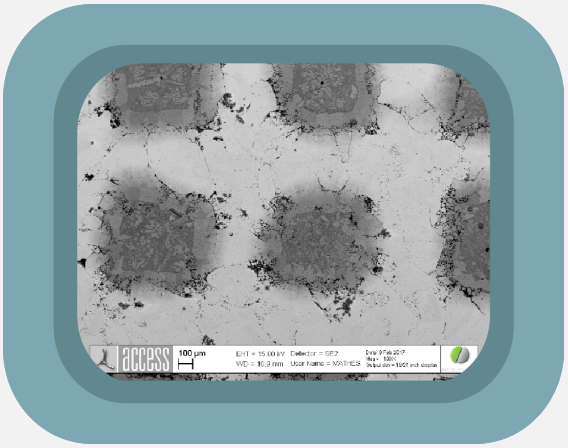


Figure 1 shows microstructure predictions after complete solidification.



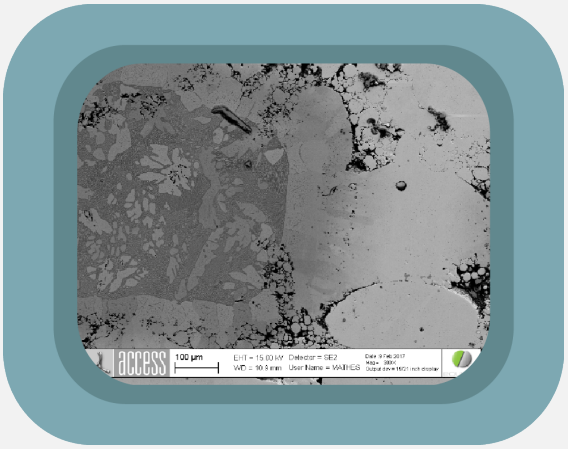


Figure 2 (top) shows a snapshot at a section of infiltrated porous Iron preforms using Iron-Aluminum melts and Figure (below) shows magnified region of the infiltrated section.

BREMBO S.p.A.

BREMBO is the world leader and acknowledged innovator of the disc brake technology for automotive vehicles. BREMBO, supplying high performance brake systems for the most important manufacturers of cars, commercial vehicles and motorbikes worldwide, has built its success on its highly innovative approach, which over the years has made it unique around the world.

Under the Equinox project Brembo will assess the applicability of the EQUINOX process to the brake system industry, also investigating its applicability for high volume products.

"Wp6- industrial demonstrators" just started with the design of moulds to especially address the correct infiltration of the porous preforms and the final product soundness. This WP will last until the end of the project aiming to manufacture and test prototypes from different industries, from aerospace to energy, automotive and hydraulics. In addition a road map to the best EQUINOX material introduction in industrial productions will be investigated.

IMDEA Materiales

In situ study of the formation of Fe-Al intermetallics during infiltration process.

Successful reactive infiltration of Al melt into Fe porous preforms have been performed for the first time using an experimental combination of x ray radiography and x ray diffraction measurements (Fig. 1). This experiment was dedicated to the investigation of the dynamics of the infiltration of Al into Fe porous preforms and the formation of intermetallic phases during the infiltration.

A designed furnace for in-situ infiltration experiments was used at EDDI beamline (BESSY synchrotron, Berlin, Germany). Radiographies and diffraction patterns are recorded for one cycle (from room temperature till cooling after heating Al up to 1000°C). Fig. 2 shows XRD patterns before and after infiltration where reflections corresponding to intermetallic phases were obtained.

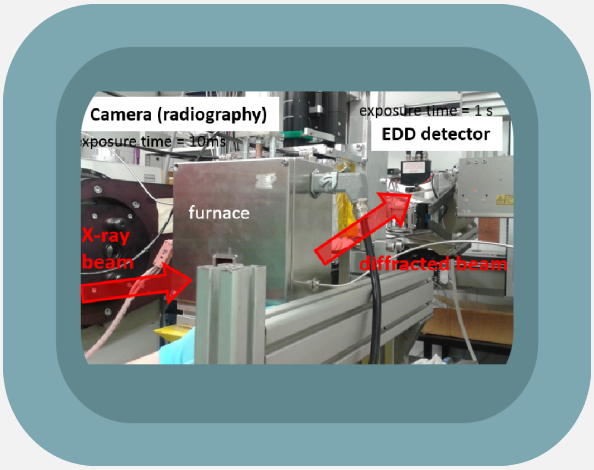


Fig. 1: Set-up of the in-situ infiltration experiment at EDDI beamline - BESSY synchrotron, Berlin, Germany

IMDEA Materiales

In situ study of the formation of Fe-Al intermetallics during infiltration process.

A radiography and optical micrograph of Fe channels infiltrated by molten Al is shown in fig. 3 (a & b). The analysis by scanning electron microscopy (SEM) confirms as well the formation of Fe-Al intermetallic phase with a tongue-like growth (Fig. 3c).The results are promising and will provide valuable data for the melt flow model and better understanding of the phase formation kinetics for Al-Fe intermetallics.Further measurements are planned at synchrotron facilities to improve the infiltration process.This work has been carried out by IMDEA partner.

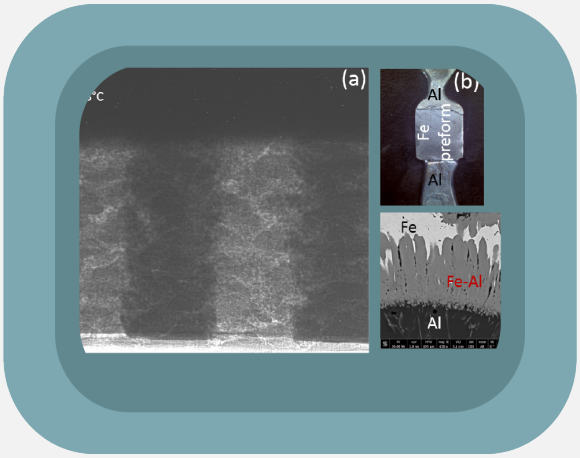
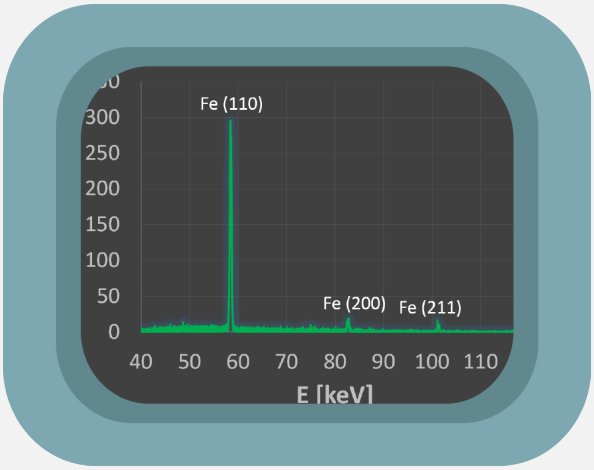


Fig. 2: (a) Radiography of 2 Fe channels (b) optical micrograph and (c) SEM micrograph after infiltration of Fe preform.



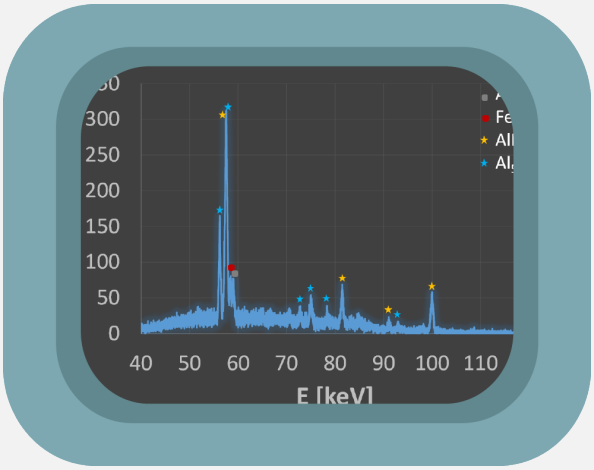


Fig. 3: XRD patterns (a) before & (b) after infiltration

National Technical University of Athens.

1. The Equinox consortium was represented at the 1st Greek Raw Materials Community Dialogue
That was held in Athens, Greece on 23 - 24 November 2016. The event was organized by EIT Raw Materials.

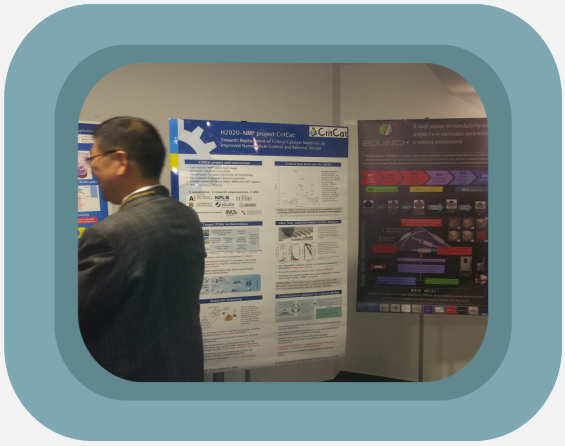


Fig. 2: Growth Raw Materials Week. Date: 28 November – 2 December 2016. Venue: Brussels, Belgium.

3. The Equinox consortium was represented in Training School "The challenge of CRMs in extreme condition: advanced multidisciplinary view" at Instituto Superior Tecnico Lisbon, Portugal on 6th and 7th February 2017

Fig. 1: 1st Greek Raw Materials Community Dialogue 23 - 24 November 2016

2. The Equinox Consortium was represented, after invitation, at the following events of the Raw Materials Week that took place at Brussels, Belgium on 28 November – 2 December 2016:
At the 6th European Union – United States of America – Japan Trilateral Conference on Critical Raw Materials (29 November) with a poster presentation.
At the Cluster & Networking event "Critical Raw Materials and Substitution" (2 December) with an oral presentation.
The event was organized by GROWTH.

Fig. 3: Training School. Date: 6th and 7th February 2017. Venue: Instituto Superior Tecnico Lisbon, Portugal.

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Photos from TUL laboratories

In the period of February-2016 to January 2017 TUL team investigated the Fe preforms before infiltration by scanning electron microscopy using secondary electron (SE) and back scaterred electron (BSE) imaging (as can be seen in figures 1,2). The porosity of preforms by means of image analysis was also studied.

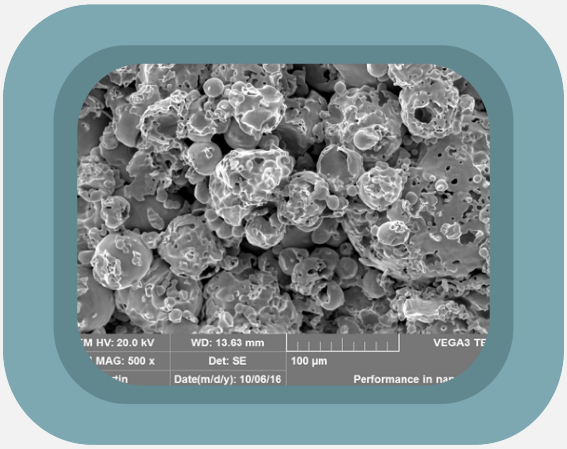

Fig. 1: TUL started first preliminary annealing experiments on the small parts of both types of received infiltrerated samples (Al and FeAl2)

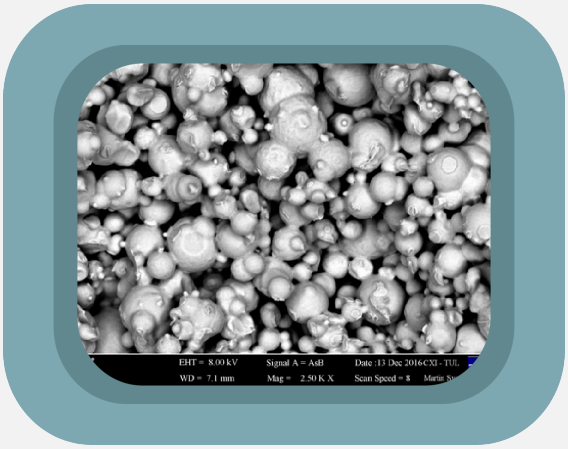

Fig. 2: The structure investigation of infiltradted samples was performed on samples made in Access. The research was focused on the samples infiltrated by pure Aluminium (casted into pre-heated Fe matrix and also casted without pre-heating) and by FeAl2. The electron microscop equipped by energy dispersive analysis was used for phase identification.

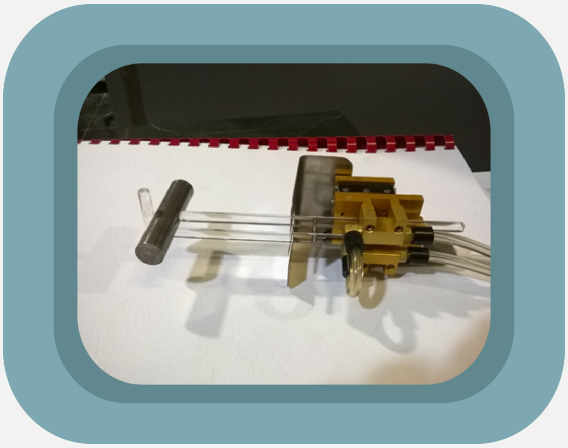

Fig. 3: TUL team investigated the Fe preforms before infiltration by high-tech experimental device Gleeble 3500. There is crossectional dilatometer for CTE evaluation in figure.

YUZHNOYE

Yuzhnoye SDO which is leading scientific and design company of Ukraine in the field of space technology development is the end user in EQUINOX Project. Under the Project YUZ provides expert assessment of applicability of the obtained material in rocket-space products operating under harsh conditions such as cryogenic temperatures and aggressive environment. The main purpose of YUZ in EQUINOX Project isadaptation of EQUINOX material to space rocketry structures. The main activity under the Project within M1-M12 was focused on production of porous iron matrixes as well as optimization of their characteristics for further infiltration with aluminum alloys.

The works were carried out in different directions, and a lot of groundwork has been done, that will be evident in the subsequent stages. From an end user perspective, we are interested toreceive manufacturing technology of the iron powder parts with properties which are not inferior to the parts made of the critical materialsand at the same time the obtained technology shall be not only of laboratory level but still successfully be applied on an industrial scale. Therefore, during Project activity, we paid close attention to the analysis of possible technologies for producing porous preforms, which will form the basis for a successful receiving of Fe-Al intermetallic compounds in future.

The main achievement is that we managed to find a relatively cheap and "user-friendly" porous iron matrix manufacturing method (press operation + sintering). Technology where parts are produced using powder metallurgy technique (solid-phase sintering) proved economically and technologically advantageous for the production of unique parts of space rocketry, in particular parts of LRE turbo pumps.

Currently we are waiting for the results of infiltration of porous-preforms made using our technology so far, but nevertheless we consider any move toward-sreducing the manufacturingcost of the new material and parts of it will allow it to adapt to a wide varietyofindustries.
Today YuzhnoyeSDO elaborates issue of utilization of iron powder of Ukrainian production
which will allow to optimize infiltration conditions due to control of porous matrixes structure.
Manufacture and delivery of the matrixes to the Project partners for further infiltration can be expected in March 2017.

YUZHNOYE

6th International Conference

The6th InternationalConference Space Technologies: Present and Future will be held from 23rdtill 26thof May, 2017inDnipro(Ukraine).The Conference is under International Academy of Astronautics (IAA)auspices.
The Conference subject is dedicated to 60 years since the launch of the first Earth's artificial satellite and to 80th years since the Birthday of Mr.Stanislav Konyukhov – distinguished researcher, engineer and designer in aerospace branch.
Basic subjects of the conference:
Section 1. Current and future space launch systems, their components and subsystems
Section 2. Current and future space satellite systems
Section 3. Future rocket engines and power propulsion systems
Section 4. Materials and technologies
Section 5. Space for humankind
The prime conference organiser is Yuzhnoye State Design Office - one of internationally renowned and top ranked scientific and design enterprises, engaged in design of rocket and space technology, which develops integrated and turnkey complicated high-tech projects in cooperation with its partners.

Fig. 1: InternationalConference Space Technologies: Present and Future May 23-26 2017 Ukraine, Dnipro

The International Conference in Dniprois an excellent opportunity to discuss new trends in space and rocket technologies, advanced development, new insight in the global space problems and rallying international concepts for their solution.The Conference is traditional and is hold biennially.
The deadline for thesis registration is 3rd of April, 2017.
For more info, please, visit the website:
http://dpukrconfiaa.org/en/

6M MEETING

Equinox project 6M meeting. Date: 12th and 13th September 2016. Venue: IMDEA Materials Institute, Madrid, Spain.

12M MEETING

Equinox project 12M meeting. Date: 14th and 16th February 2017. Venue: ACCESS EV, Aachen, Germany..

More info on Equinox-project

www.equinox-project.eu

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