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High-Temperature Properties of MIM-Processed Superalloys

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1 Abstract

Two Ni-based PM superalloys (Inconel 713C & Udimet 720), which have been processed by metal injection moulding (MIM), were analyzed with respect to their high-temperature properties. This included tensile tests at temperatures up to 900°C and evaluation of oxidation resistance up to 1100°C. The first test series lead to promising results when compared to other processing routes; however, optimizations concerning impurity contents are necessary to improve the high-temperature performance.

2 Introduction

There is a continuous demand for shifting the operating temperatures to higher values, especially in the automotive and aircraft industry, combined with the need of cost-effective mass production of the respective parts. With respect to suitable high-temperature materials, these are subject to complex thermomechanical loads at service temperatures. One of the most promising material class to fulfil all the requirements are Ni-based superalloys, which have been focussed on in R&D as well as in production of the respective parts for many years due to their excellent combination of mechanical strength and corrosion resistance.
Regarding cost-effective production routes, the metal injection moulding (MIM) process offers an advantageous route for production of large numbers of near-net-shape parts. Therefore, MIM-processed superalloys are one of the most promising candidates for next-generation parts in high-temperature applications.
There is knowledge available on how to process superalloys by MIM and their room-temperature properties [1, 2]. However, in order get a more profound understanding on how these MIM-processed alloys are going to perform at temperatures in the range of actual service conditions, their high-temperature properties need to be analyzed. This study has the aim of providing first results for selected alloys.

3 Experimental

Two Ni-based PM alloys (Udimet 720 and Inconel 713C) were evaluated for their properties at elevated temperatures. These alloys contain significantly more Al (up to 6 wt%) than previously used MIM-materials (typical compositions see Table 1). Therefore, they are able to form γ' precipitates, which lead to an increase in e.g. tensile and creep strength at high temperatures [3].

	Ni	Cr	Al	Co	Mo	Ti	W	Nb
U720	bal.	18	2,5	15	3	5	1,25	
IN713C	bal.	12,5	6,1		4,2	0,8		2

Table 1: typical chemical composition of studied superalloys (wt%)

Samples were provided by Schunk Sintermetalltechnik GmbH as MIM tensile bars (Figure 1). Both alloys underwent a HIP treatment after the MIM process. Furthermore, Udimet 720 was subjected to an additional heat treatment in order to adapt the morphology and size of the precipitates (parameters see Table 2). As Inconel713 is used in the "sintered+HIP" condition, no heat treatment of this kind was applied for this alloy.

alloy	HIP	heat treatment
Inconel 713C	1200°C, 100 MPa, 4h	---
Udimet 720	1130°C, 140 MPa, 4h	solution anneal: 1100°C, 1h ageing: 650°C, 24h + 760°C, 16h

Table 2: post-processing of studied alloys

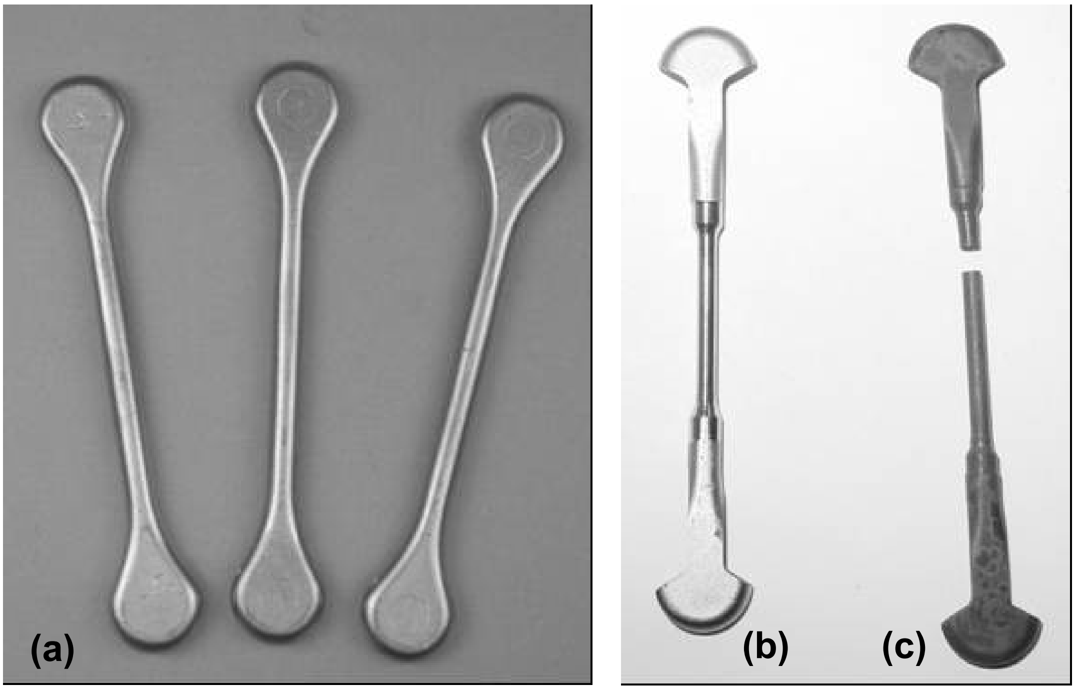


Figure 1: (a) MIM tensile bars, (b) MIM tensile bar, machined for HT testing, (c) MIM tensile bar after HT mechanical test (tensile test)

Mechanical testing was done using a Zwick universal testing machine 1476. This machine is equipped with a furnace (HTO-08, Maytec), which operates under air, and a high-temperature extensometer (PMA-12/V7/1, Maytec). Prior to testing, the MIM samples were machined in order to fit into the sample holder. The assembly was then heated to the respective target temperature, which was in the range of 650°C – 900°C, at a rate of 15 K/min. In order to ensure a homogeneous temperature along the sample, a holding time of 60 min was inserted before the actual test started. For the tensile test, the machine speed was 0,05 / min and the test was finished when the force dropped below 80% of its maximum value.

The oxidation resistance was analyzed by different tests under air. In one isothermal test, samples were exposed to air at temperatures between 900 and 1100°C for up to 100h. Additional analysis included gas analysis for impurities (carbon, oxygen, nitrogen), SEM for microstructure, SEM-EDX for composition and element distribution,

4 Results and discussion

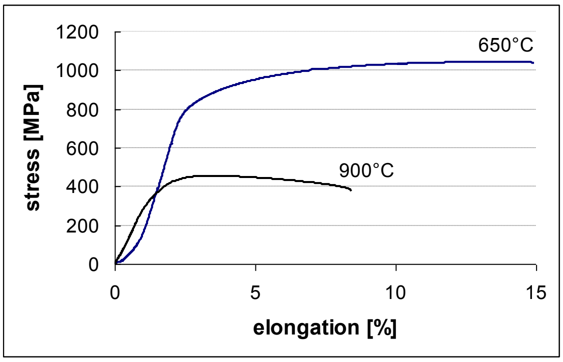
Density & impurity contents

Density measurements using the Archimedes method indicate that all samples are fully densified (> 99% of theoretical density). Typical impurity contents for both alloys are summarized in Table 3.

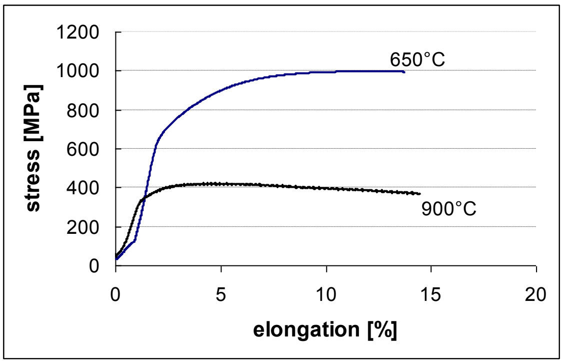
alloy	C (%)	N (%)	O (%)	remarks
Inconel 713C	0,32	0,02	0,25	C = 0.17% , N = 0.02%, O = 0.04% [1]
Udimet 720	0,09	0,002	0,26	C = 0.02% [8] C = 0.04% [9] C = 0.01 – 0.04 % [10]

Table 3: impurity contents (carbon (C), nitrogen (N) and oxygen (O)) for MIM samples in „as-sintered" condition; values from literature are given for comparison

Hot tensile test



Inconel 713C



Udimet 720

Figure 2: typical stress-strain curves at elevated temperatures for alloys of this study

Figure 2 shows typical stress-strain curves for both alloys in the temperature range 650 – 900°C. Values for the yield strength $R_{p0.2}$ and the tensile strength R_m are given in Table 4. By comparing the values of Inconel 713C with those obtained by investment casting [4] and on cast samples [5] it can be concluded that the MIM samples yield comparable values at 650°C. However, at the test temperature of 900°C, the tensile properties obtained for the MIM samples are lower.
For alloy Udimet 720 tensile properties are inferior to previous tests on P/M [10] and cast samples [6] (see also Table 5 for additional values at other temperatures).

alloy	T [°C]	$R_{p0.2}$ [MPa]	R_m [MPa]	remarks
IN713	650	800	1043	$R_{p0.2}$ = 650 – 670 MPa [4] $R_{p0.2}$ = 550 – 850 MPa [5] R_m = 660 – 940 MPa [5]
IN713	900	373	455	$R_{p0.2}$ = 400 MPa [4] $R_{p0.2}$ = 320 – 460 MPa [5] R_m = 600 – 780 MPa [5]
U720	650	677	1000	$R_{p0.2}$ = 1050 MPa [6] $R_{p0.2}$ = 880 – 1103 MPa [10], depending on heat treatment R_m = 1367 - 1476 MPa [10], depending on heat treatment
U720	800	595	656	$R_{p0.2}$ = 880 MPa [6]
U720	900	343	390	

Table 4: tensile properties of MIM-processed alloys Inconel 713C & Udimet 720 at elevated temperature; values from literature are given for comparison

T [°C]	$R_{p0.2}$ [MPa]	R_m [MPa]	remarks
600	675 - 830	1190 - 1290	[7], values depend on grain size
700	705 - 824	800 - 1225	[8], values depend on test speed
704	690 - 850	1040 - 1190	[9], values depend on heat treatment
760	780 - 1069	1103 - 1180	[10], depending on heat treatment

Table 5: high-temperature tensile properties of alloy Udimet720 at temperatures other than those investigated in this study

The most likely reasons for the differences in tensile properties are the following:

(1) high carbon content for both alloys: Table 3 shows that the C-values are substantially higher for this first series of MIM samples and also above specification values (IN713C [1], U720 [11]). Figure 3 and Figure 4 show that substantial amounts of carbides are formed. Although carbides on grain boundaries contribute beneficially to high-temperature strength [3], increased amounts of high-temperature stable carbides on grain boundaries and inside grains can have detrimental effects on tensile properties by e.g. being initiation sites for cracks [12, 13, 14, 15]

(2) high oxygen content for both alloys: this can lead to formation of e.g. unwanted oxides, which have similar detrimental effects on tensile properties as carbides

(3) heat treatment parameters (U720): effects on high-temperature mechanical properties are reported: [10, 16], also a beneficial effect on strength is found by modifying the precipitate morphology and size distribution [7, 17]

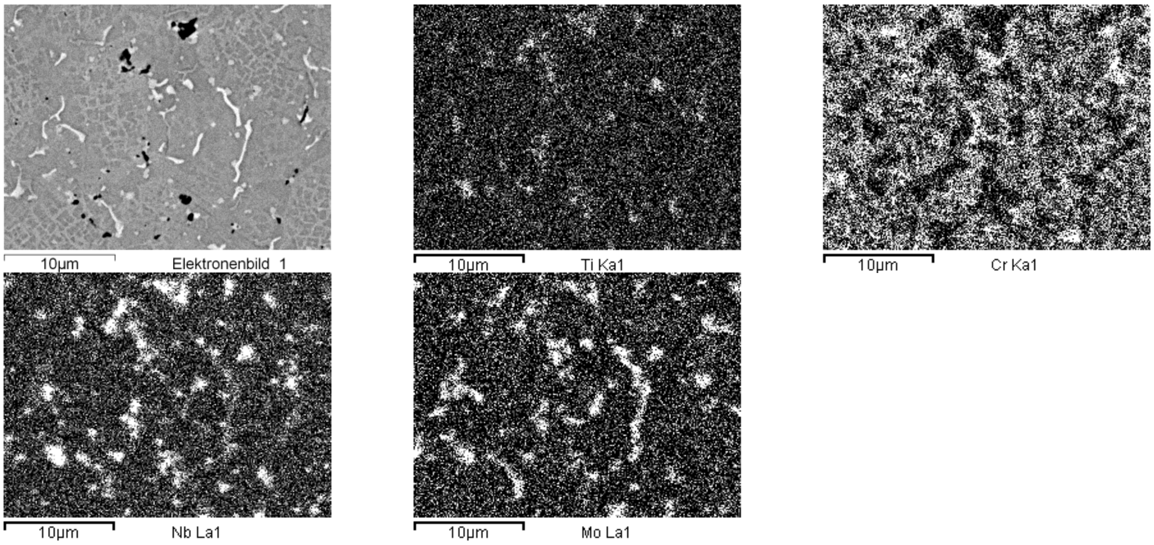


Figure 3: element distribution (Ti, Cr, Nb, Mo) in MIM sample of alloy IN713C (condition as sintered)

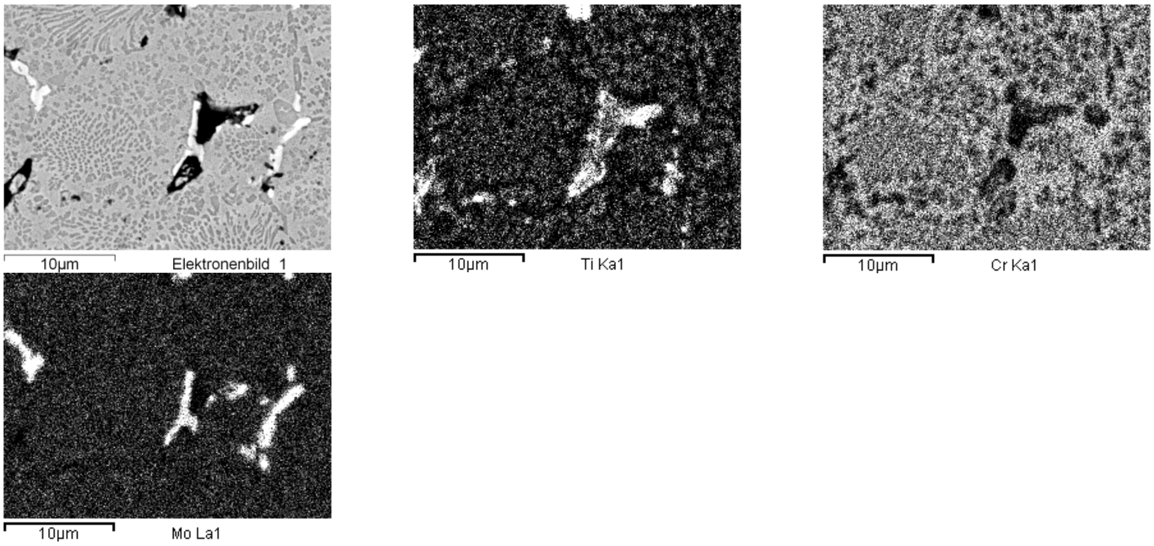


Figure 4: element distribution (Ti, Cr, Mo) in MIM sample of alloy U720 (condition as sintered)

oxidation resistance

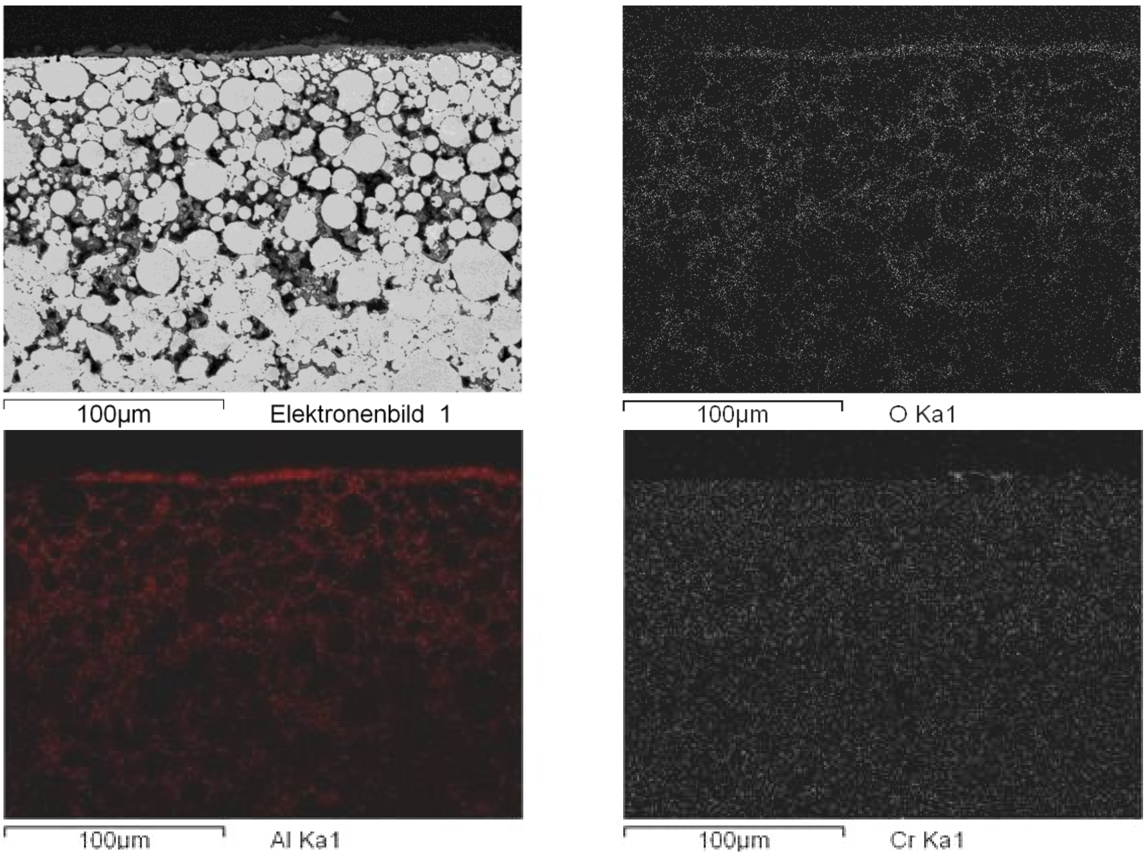


Figure 5: element distribution in alloy IN 713C after isothermal oxidation (900°C, 100h, air)

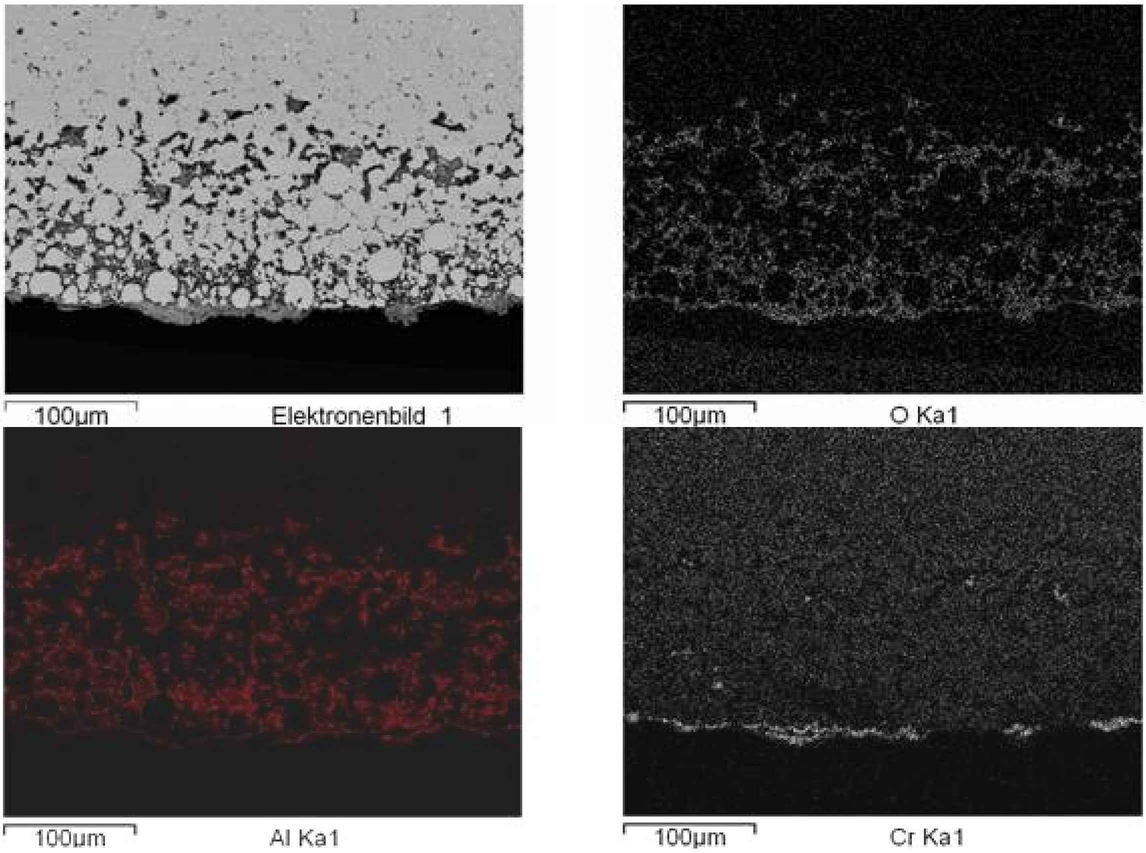


Figure 6: element distribution in alloy IN 713C after isothermal oxidation (1000°C, 100h, air)

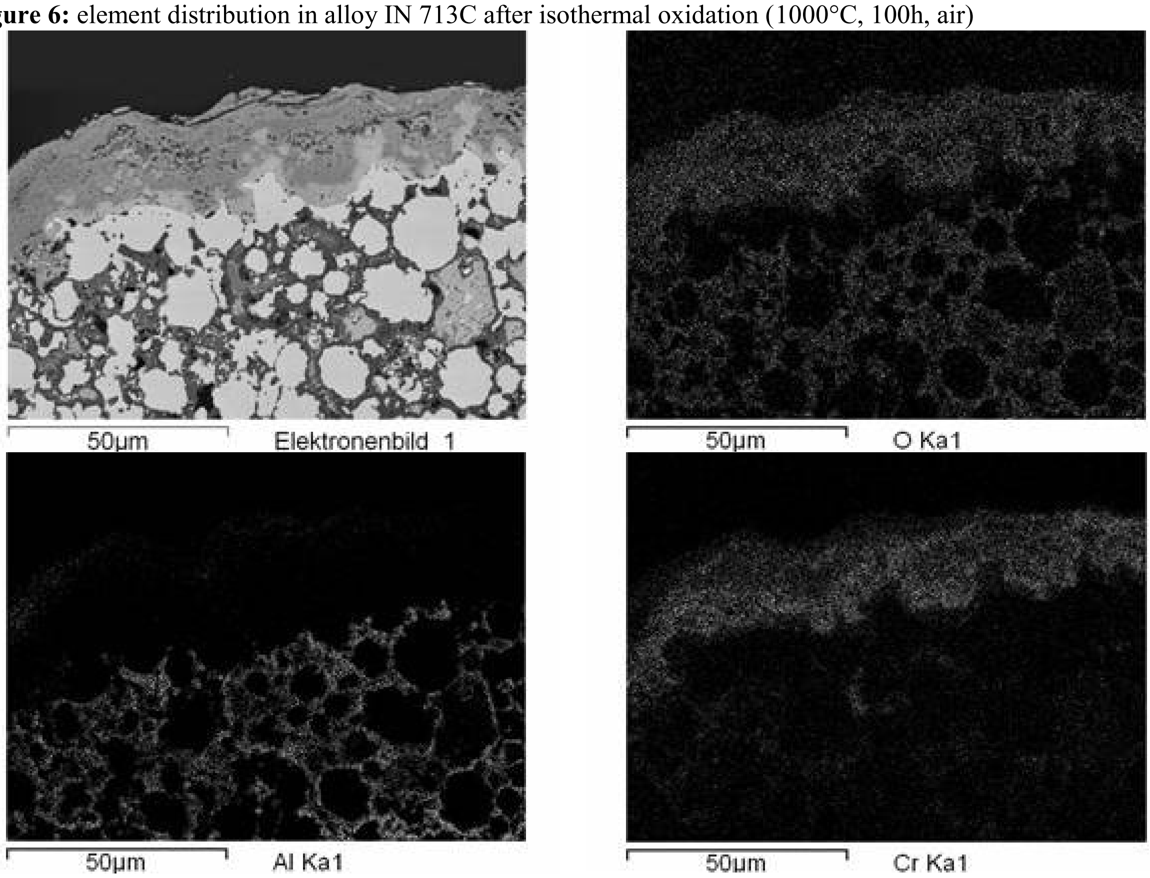
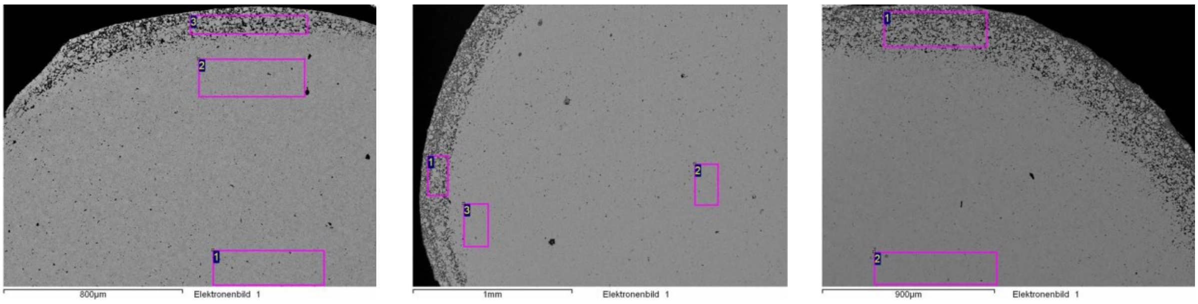


Figure 7: element distribution in alloy IN 713C after isothermal oxidation (1100°C, 100h, air)



900°C 1000°C 1100°C

Figure 8: SEM images of alloy IN 713C after oxidation for 100h at different temperatures

Figure 5 - Figure 7 show the element distributions at the edge of a MIM-bar (alloy IN 713C) after isothermal oxidation between 900 - 1100°C for 100h. All samples are in the as-sintered condition. In this temperature range, Al_2O_3 as well as Cr_2O_3 can form as protective oxide scales. At 900°C, alumina is pronounced, while at 1000°C and 1100°C both oxides form. Figure 8 shows overview images of the edge up to the middle region of oxidized MIM bars (alloy IN 713C). Based on these images the thickness of the overall oxide scale (i.e. the closed scale on top and the porous oxidized region below) can be estimated: it increases with temperature from 136µm (900°C) up to 264µm (1100°C). Below the scales and the porous edge region no further internal oxidation takes place. Therefore it is stated that the alloy IN 713C is able to passivate under oxidizing conditions.

5 Conclusions

A test series has been started on alloys Udimet 720 and Inconel 713C, which were processed by MIM, in order to evaluate their high-temperature performance. First tests included tensile tests up to 900°C and isothermal oxidation tests up to 1100°C.
Concerning high-temperature strength, IN 713C shows a high potential compared to other processing routes. U720 however needs improvement with respect to mechanical strength.
It is concluded that in order to increase the high-temperature tensile strength, the primary effort needs to be the significant reduction of impurities, namely carbon and oxygen. This can be achieved by optimizing the thermal debindering regime. Current tests with new regimes show indeed that reductions down to the level of impurities in the alloy powders are possible. Furthermore, new powder variants with reduced carbon contents (Inconel 713LC & Udimet 720Li) will be included in subsequent tests. Concerning high-temperature oxidation resistance, first tests under isothermal conditions indicate a good performance of alloy IN 713C. Further tests (thermogravimetry, thermocycling conditions) on both alloys will help to get a better understanding of the overall high-temperature performance of these Al-containing MIM-superalloys.

6 References

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