There is a linear dependency between r³ and time, which confirms the expected diffusion - controlled Ostwald ripening process. Then, equation (2) can be written with r³ = 3 as r³(t)=r³(0)+K't, where r(0) is the particle size at the start of coarsening and Figure 6b shows that it is 2±1 and 7±2 nm for smaller and larger precipitates, respectively.

Hardening phase precipitation study in the new VDM® Alloy 780 by *in-situ* high temperature neutron scattering techniques

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Introduction

The new Ni-base superalloy VDM® Alloy 780 has been developed to reach improved mechanical properties at high temperature that would allow working temperatures up to 750 ºC. [1,2] The structure of this new superalloy is formed by the austenitic matrix (γ phase) strengthened by nano-sized intermetallic precipitates of Ni_3Al (γ') and high temperature stable precipitates composed of the Ni_3Nb-based (δ) and Ni_3Ti-based (η) phases. [3-5]

The γ' hardening precipitates influence the mechanical properties, therefore their volume fraction, size and morphology has to be investigated and optimized in order to obtain improved mechanical properties at high temperature operation conditions. For this, it is required a) a proper understanding of the precipitation process of the γ' hardening phase, which will allow adjusting the multi-step heat treatments of the precipitation; and b) study of the hardening precipitates evolution with temperature and time, which will allow the understanding of the growth/coarsening of the γ' precipitates, to ensure the long-term characteristics of the material.

This study shows the precipitation behavior of the γ' hardening phase in the VDM® Alloy 780 by means of in-situ small-angle neutron scattering (SANS) and time-of-flight (TOF) neutron diffraction (ND) at high temperatures and Atom Probe Tomography (APT).

VDM® Alloy 780 Composition

	Ni	Co	Cr	Fe	Mo	Nb	Al	Ti
wt.%	balance	25	18	<3	3	5.4	2	0.2

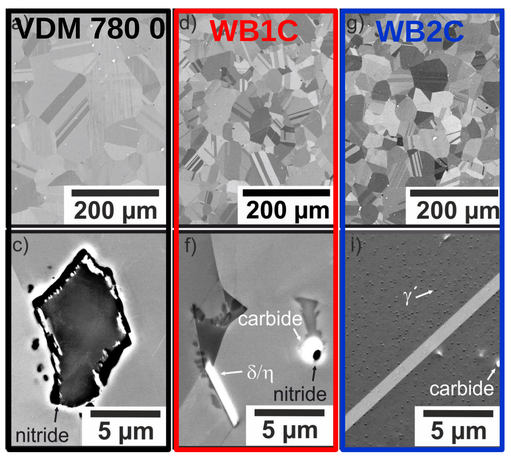
Heat treatments

Performed at different points of the precipitation process

VDM 780 0	VDM 780 WB1C	VDM 780 WB2C
1020°C/1h/AC + 1080°C/1h/WC	1020°C/1h/AC + 1000°C/0.5h/FC 1 K/min + 975°C/16h/WC	1020°C/1h/AC + 980°C/1.5 h/WC + 720°C/8h/FC 50 K/h+ 620°C/8h/AC

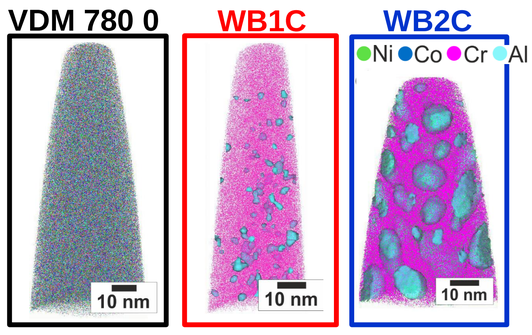
Structural characterization of the new developed VDM® Alloy 780 at RT

SEM



- **VDM 780 0**: γ matrix (66 µm grain size) + TiNb- carbides and nitrides.
- **VDM 780 WB1C**: γ matrix (35 µm grain size) + TiNb- carbides and nitrides.
- **VDM 780 WB2C**: γ matrix (33 µm grain size) + small γ' hardening precipitates + TiNb- carbides and nitrides.

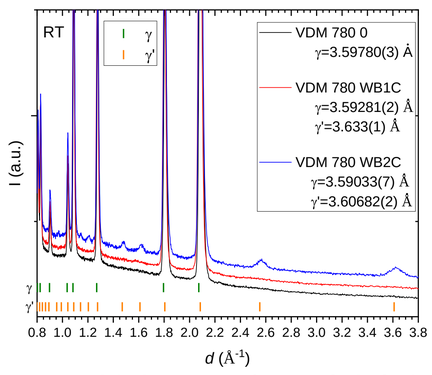
APT



APT composition (at.%)

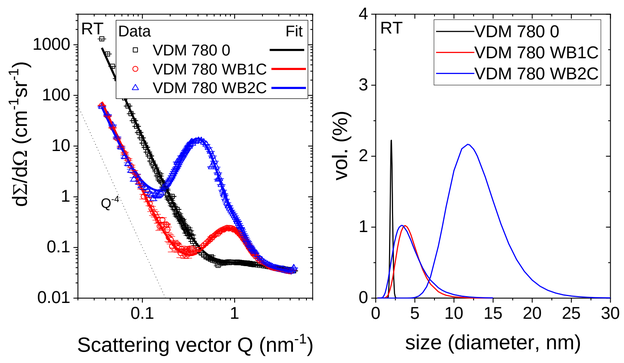
		Ni	Co	Cr	Nb	Al	Ti	Mo	Fe
0	γ	44.8	24.3	19.4	3.5	4.9	0.4	1.9	0.7
WB1C	γ	44.9	24.0	20.0	3.1	4.5	0.5	2.0	0.9
	γ'	44.7	23.9	19.8	3.5	4.9	0.3	2.0	0.8
WB2C	γ	34.7	31.2	28.2	1.4	0.1	2.4	0.9	1.1
	γ'	64.3	9.9	3.4	6.9	13.1	0.8	0.9	0.0

TOF ND



- **VDM 780 0**: γ matrix + NbC (<0.1wt.%).
- **VDM 780 WB1C**: γ matrix + γ' phase (4.6 wt.% and 4 nm) + NbC (<0.1wt.%).
- **VDM 780 WB2C**: γ matrix + γ' phase (21.8 wt.% and 12 nm) + NbC (<0.1wt.%).

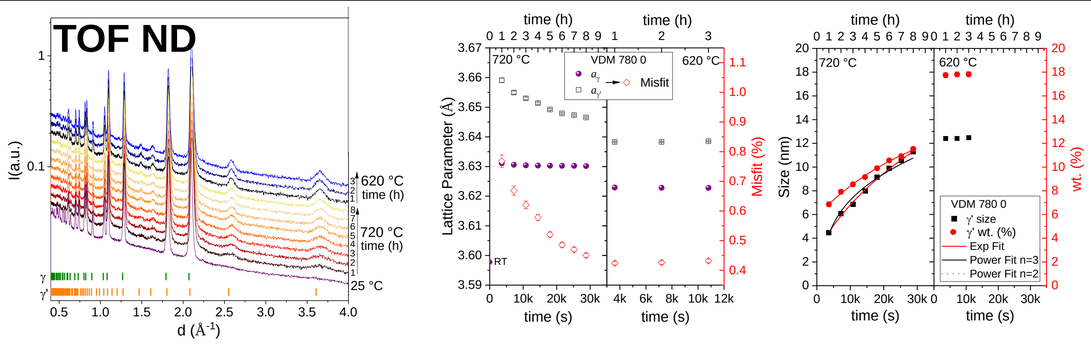
SANS



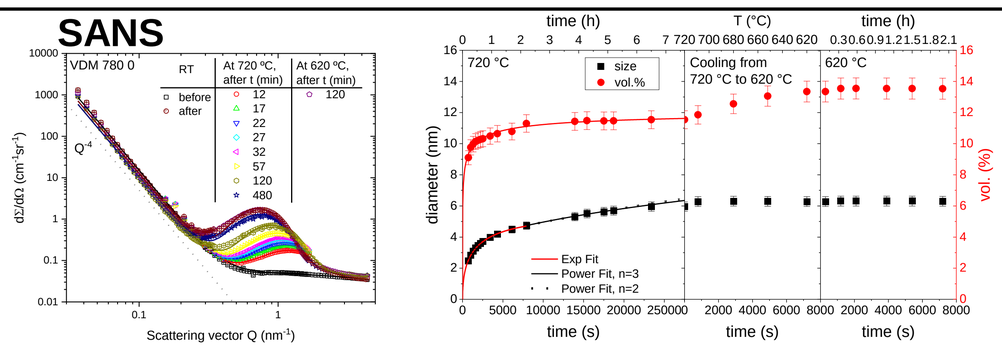
- **VDM 780 0:** large-scale objects and/or defects in the matrix + one size distribution of γ' precipitates (2 nm – 0.6 vol.%).
- **VDM 780 WB1C:** one size distribution γ' (4 nm – 3.5 vol.%).
- **VDM 780 WB2C**: two size distributions γ' (4 nm – 3.5 vol.% + 13 nm – 14 vol.%).

High T precipitation of the γ' phase by in-situ TOF ND (GEM, ISIS) and Small-Angle Neutron Scattering (SANS-1, MLZ)

VDM 780 0



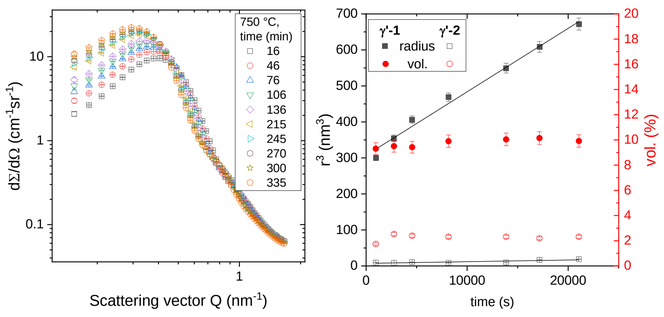
- Precipitates nucleation at 720 ºC: exponential growth of wt.% with Avrami's parameter n = 1.2 → decreasing nucleation rate. Size increase following an exponential growth or diffusion-controlled growth $d^2 = d^2(0) + Kt$, with $d(0)$= 2 nm and K=4x10^{-3} nm^2/s.
- At 620 ºC there is no more precipitation or coarsening.



- At 720 ºC there is an exponential increase of vol.% and size (before 2 h) as a function of time: vol.% indicates nucleation of precipitates (Avrami theory) and that nucleation is the limiting step before 2 h. After 2 h size grows following $d^n = d^n(0) + Kt$, with n=2 or 3 → growing and coarsening of precipitates with $d(0)$= 4 nm and K=1x10^{-3} nm^2/s or 8x10^{-3} nm^3/s.
- At 620 ºC there is no more precipitation or coarsening.

Precipitates evolution at operation temperature, 750 ºC

VDM 780 WB2C - SANS



- Diffusion - controlled Ostwald ripening process: $r^3 = r^3(0) + K't$
- Particle size at the start of coarsening $r(0)$= 2 nm and 7 nm for smaller and larger precipitates, respectively.
- Coarsening rate at 750 ºC, K' = 0.017 nm^3/s, slower than other reported Ni-alloys (0.104 nm^3/s for Inconel 718).

Conclusions

- APT provides composition information necessary for the scattering contrast calculation that will allow vol.% determination from SANS measurements.
- In-situ SANS at high temperature allows following the γ' precipitation during the precipitation steps at 720 and 620 ºC:
  a) at 720 ºC → nucleation of precipitates follows Avrami theory, limiting step up to 2 h and then growing and coarsening of the precipitates.
  b) at 620 ºC →no new precipitation or precipitation coarsening.
- In-situ SANS at operation conditions shows diffusion-controlled Ostwald ripening process of the γ' precipitates with a coarsening rate at 750 ºC, K = 0.017 nm^3/s, slower than other reported Ni-alloys (0.104 nm^3/s for Inconel 718).

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