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Tensile properties of a non-line-of-sight processed β - γ - γ' MCrAlY coating at high temperature

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ABSTRACT

The tensile properties of a NiCoCrAlYTa coating manufactured by the Tribomet® process were examined in a wide range of temperatures representative to turbine in-service conditions. Thin and bulky freestanding coating specimens (FSCS) were tested and compared in order to evaluate the mechanical strength and ductility of this material. Comparable results were found for both the specimen geometries, i.e. a brittle-to-ductile transition temperature (BDTT) between 650 and 700 °C, a significant increase of the ductility (up to 5.5% at 800 °C) and a drop of the tensile strength above the BDTT. Thin FSCS demonstrated slightly lower yield strength and ultimate tensile stress compared to bulky FSCS. Fractographic examinations aimed to suggest that this difference in mechanical properties was partly attributed to a lesser cohesion and brittle behavior of a β -rich layer found in the extreme surface of the overlay coating due to the deposition technique. This poor local mechanical strength is believed to be detrimental for the integrity of thermal barrier coatings (TBC) systems and attentions in the design of such functionally graded materials have to be paid to prevent early in-service damages.

1. Introduction

MCrAlY coatings are generally used in the design of turbine blades and vanes to protect the thin-walled airfoil from high temperature oxidation and hot corrosion (type I and II) [1,2]. These coatings are particularly encountered in aeronautic and power plant applications as a final environmental barrier against severe atmospheric conditions. They found also applications as bond coatings in thermal barrier coating (TBC) systems applied to shrouds, transition ducts and combustion liners in addition to turbine blades and vanes [3,4]. The chemical composition of such coatings has been optimized through decades to form adherent thin- and low-growth-rate oxides (α -Al₂O₃ - commonly denoted thermally grown oxide (TGO) - for high temperature oxidation protection and Cr₂O₃ for intermediate temperature corrosion). The addition of reactive elements [5–7] and/or platinum [8,9] strongly improved the efficiency of coatings under thermal cycling stresses, limiting the oxidation rate of the so-called TGO and thus its early spallation. On the other hand, blades and vanes are mostly subjected in service to high temperatures, high-speed rotation and high temperature

transient. Under such in-service stresses, damages initiating within the coating or at coating interfaces were reported in the literature to contribute to the early failure of the coated components [10–14]. While the selection of those coatings is generally based on its primary-function properties, i.e. its oxidative and corrosive properties under isothermal and cyclic stresses, the mechanical and physical properties of the coatings have a significant impact on the integrity of those multi-layered materials. The chemistry of the coating and the deposition process need to be tailored to limit the extent of the interdiffusion zone, locally affecting the original properties of the substrate, i.e. the single crystal Ni-based superalloy [15]. Moreover, the mechanical and thermal properties mismatches between the substrate and the coating have to be adapted to overcome these premature damage mechanisms. Ductile and creep-resistant coatings are better suited to accommodate thermal mismatch stresses, especially in the range of operating temperatures. Well known to be particularly brittle at low temperatures, MCrAlY coatings exhibit a higher ductility above a given temperature denoted the brittle-to-ductile transition temperature (BDTT). Damage-tolerant design of coated components preferentially deals with the BDTT than the effective ductility of the MCrAlY coating at given temperatures since this latter property is particularly difficult to assess, both in freestanding coating or multi-layered specimen approaches. As far as the BDTT is concerned for thermal cycling applications, the lower the better. BDTT of MCrAlY coatings was found to be sensitive to the chemistry and its microstructure, the strain rate and the thickness of the coating for coated components [16–18]. On the one

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hand, the aluminum content strongly affects the occurrence and volume fraction of the β -NiAl phase, responsible of the brittleness of the coatings [3,4,19–23]. Chromium and platinum addition also tend to decrease the ductility of the coating. On the other hand, chemical elements prone to form γ -Ni phase tend to increase the ductility of such materials. Moreover, thinner coatings and lower strain rates lead to an increase of the ductility [24]. Therefore, the thickness of those coatings has to be optimized in order to efficiently comply with i) oxidation/corrosion degradation, and to limit, ii) the “none-load bearing” mass at high temperatures and iii) the brittleness and the crack sensitivity at intermediate/low temperatures.

BDTT values reported in the literature on coatings are not straightforwardly comparable due to measurement criteria variable from a study to another one, either with freestanding coating samples or multi-layered samples. Depending on the study, BDTT was measured to be the temperature at which i) the first crack appears at 1% deformation [25], ii) the ductility increases while the mechanical strength (yield stress (Y.S.) and ultimate tensile strength (U.T.S) decrease [25,26]), iii) the elongation-to-the-first-crack occurrence rapidly increases [25–27], iv) the hardness value clearly drops [23,28]. Various methods to detect the appearance of the first crack during in-temperature tensile or punch tests [21,29] were proposed, i.e. observations after interrupted tensile tests [11], continuous measurement of the potential difference [25] and continuous measurement of the acoustic emission [18].

The mechanical strength and the ductility of the coating might be much more valuable inputs for predictive numerical models than BDTT of the thermomechanical behavior and in-service durability of multilayered materials. However, experimental characterizations tools dedicated for the mechanical testing of tens-to-hundred micrometer thick samples at high temperature mostly failed in the measurement of the material ductility. Recent development in high temperature micromechanical test rigs aimed to better assess ductility via thin free-standing specimens [30]. In the present study, the tensile and thermal expansion behavior of thin and bulky NiCoCrAlYTa obtained with the Tribomet® process from PRAXAIR Surface Technologies will be investigated via this recent technique and conventional test rigs. The Tribomet® process is a non-line-of-sight deposition and low residual stress/distortion process, making it attractive for homogeneous coating (thickness, composition and microstructure) on complex geometry components as well as no distortion requirements [31]. However, the surface integrity (roughness, composition, microstructure) of such process is important for as-deposited applications, especially in the case of TBC systems. Such surface/sub-surface local properties might trigger an early failure due to crack initiation from the surface or due to spallation of the ceramic top-coat at the coating/TGO interface for coating and TBC systems applications [13], respectively. Therefore, this surface integrity aspect will be investigated via the comparison of properties between bulky polished and thin as-received specimens and fractographic analyses.

2. Experimental materials and procedures

2.1. Materials

NiCoCrAlYTa overlay coatings were deposited on substrates with the Tribomet® process from PRAXAIR Surface Technologies. This process consists in an electro-deposition of CrAlYTa particles entrapped in a Ni,Co matrix [27]. According to the deposition parameters, the nominal composition (at. %) of the coating was Ni–19.8Cr–17.2Co–0.2Mo–0.5W–17.2Al–0.1Ti–1.3Ta–0.8Y. Two different coating thicknesses (bulky: $350 \pm 25 \mu\text{m}$ and thin: $70 \pm 5 \mu\text{m}$) were chosen in order to compare the tensile properties of bulky and thin freestanding coating specimens (FSCS).

Bulky coatings were deposited on aluminum plates, this latter being chemically removed to avoid contamination from the factice substrate. The preparation of the thin and bulky FSCS is described in a following

section (2.3.1). Thin coatings were deposited on Ni-based single crystal superalloy MC2 plates previously solution heat-treated ($1300 \text{ }^\circ\text{C}$ -3 h).

Diffusion heat treatments ($1080 \text{ }^\circ\text{C}$ -6 h + air cooling followed by $820 \text{ }^\circ\text{C}$ -20 h + air cooling) were then performed under vacuum on the thin-coated Ni-based single crystal superalloy and on the bulky free-standing coatings. The diffusion heat treatments aimed to ensure a mechanical cohesion between the particles and the matrix due to interdiffusion and the β - γ - γ' microstructure. This metallurgical state was denoted “as-received” state.

2.2. Characterization techniques

Microstructural observations and fractographic analyses were performed using a LEO435VP scanning electron microscope (SEM) in a backscattered electron mode and secondary electron mode, respectively. EDX analyses were conducted with a JEOL JSM6400 SEM equipped with an Oxford EDAX analyzer in order to identify the different phases found in the as-received coating. Probe current was first measured through a Faraday cage in order to guarantee a 1.5 nA current. The calibration of each constituent was carried out on real standards.

2.3. Mechanical characterization of bulky and thin FSCS

2.3.1. Specimen preparation

In the present study, bulky and thin FSCS were investigated and different specimen geometries were used for specific mechanical tests, as shown in Fig. 1.

For bulky FSCS, different flat specimen geometries were used in order to perform resonant dynamic measurements (Fig. 1.a) and conventional tensile tests (Fig. 1.b). As previously mentioned, bulky coatings were extracted from aluminum substrates via chemical etching then heat treated. For resonant dynamic measurements, ribbon-shape samples were cut via a precision cutting machine with the following dimensions (15 mm long, 3 mm wide and 0.3 mm thick in Fig. 1.a). A 1- μm tolerance geometrical tolerance of the thickness was required for the accurate determination of the dynamic elastic modulus ($\pm 1 \text{ GPa}$). For

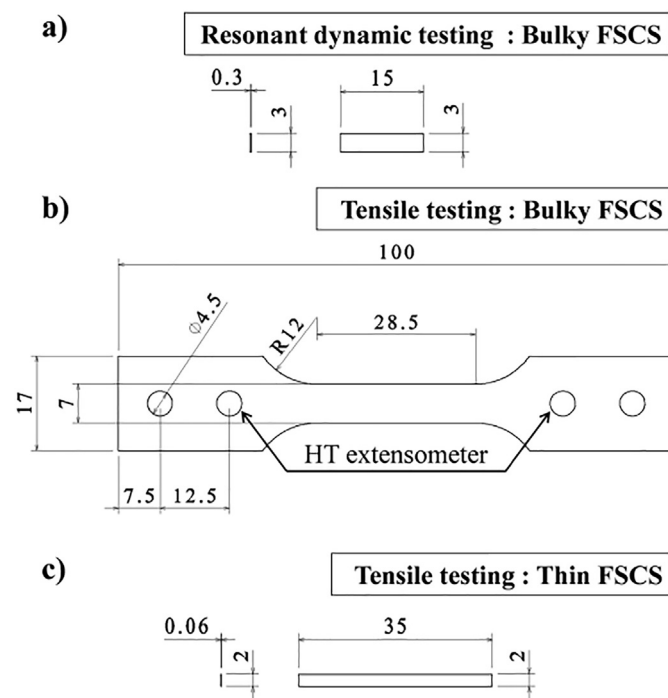


Fig. 1. Different specimen geometries used for the mechanical testing of bulky and thin FSCS: a) Resonant dynamic testing on bulky FSCS, b) Tensile testing on bulky FSCS, and c) Tensile testing on thin FSCS.

conventional tensile tests, specimens were CNC machined to obtain the geometry shown in Fig. 1.b. Edges were gritted with SiC grit paper (up to P4000) in order to remove any small cracks produced during the machining operation of this room temperature brittle material. All the samples were finally gritted then polished with a precision jig on the two faces down to a 0.25 μm diamond particles finishing to entirely remove the rough outer layer resulting from the deposition process. The average thickness of the bulk FSCS was about $300 \pm 5 \mu\text{m}$.

Thin FSCS for tensile tests were extracted from the coated Ni-based single crystal superalloy MC2 plates. Ribbon-shape specimens were first sliced via a STRUERS Secotom-50 precision cutting machine to extract 35 mm long and 2 mm wide specimens of the coated superalloy system. The top surface of the thin coating was polished in order to slightly smooth the initial roughness inherent to the Tribomet® process to an acceptable industrial roughness ($R_a < 2 \mu\text{m}$ and $R_z < 6 \mu\text{m}$). The substrate and the interdiffusion zone were then gritted in order to only extract a tens-of-microns-thickness NiCoCrAlYTa FSCS. To achieve this FSCS extraction, a PP5GT Logitech precision jig was used during the gritting/polishing operations in order to minimize the stress introduction due to the mechanical polishing and to ensure parallelism on such long and thin specimens. The detailed procedure for the thin FSCS preparation was fully described in a recent paper [30]. The average thickness of the thin FSCS finally ranged from 54 to 60 μm and the variation in thickness was about $\pm 0.5 \mu\text{m}$ along the specimen gage. The thin FSCS geometry is depicted in Fig. 1.c.

2.3.2. Tensile characterization for bulky NiCoCrAlYTa FSCS

In the present study, the elastic properties of the bulky NiCoCrAlYTa coating were measured at various temperatures via a resonant dynamic technique following ASTM E 1876-15 standard. The dynamic elastic modulus was obtained via measuring the out-of-plane flexure of the bulky FSCS under frequency continuous excitation [32]. The excitation and detection were insured by an electrostatic device (capacitance created between the sample and a unique electrode). Measurements were performed every 50 $^\circ\text{C}$ from room temperature up to 1000 $^\circ\text{C}$ with a principal bending mode resonating between 4.7 and 3.5 kHz at temperatures ranging from 20 $^\circ\text{C}$ to 1000 $^\circ\text{C}$, respectively. Tests were performed under a secondary vacuum (10^{-6} mbar) in order to limit surface reactivity of the specimen at high temperatures. The dynamic elastic modulus, $E(T)$, was calculated from the bending resonance frequency F via Eq. (1) at each temperature increment.

$$E(T) = 0.9464\rho F^2 \frac{L^4}{h^2} K \left(\frac{h}{L}, \nu \right) \quad (1)$$

where L , h , ρ and $K(h/L, \nu)$ are the length, the thickness, the density and a correction factor close to 1 [33], respectively.

Tensile tests on bulky NiCoCrAlYTa FSCS were conducted with an INSTRON 100-01 conventional tensile machine using a radiative furnace under high purity argon flow at room temperature, 650, 750 and 850 $^\circ\text{C}$. A constant crosshead displacement rate was applied during the tensile test to ensure a strain rate of $5 \times 10^{-5} \text{ s}^{-1}$ up to failure. A high temperature extensometer positioned in the holes next to the gage of the bulky FSCS was used to assess macroscopic deformation (Fig. 1.b). The initial gage length for the calculation of the deformation was assumed to be the one machined, i.e. 28.5 mm.

2.3.3. Tensile characterization for thin NiCoCrAlYTa FSCS

Tensile tests on thin FSCS were conducted using a recently developed test rig at CIRIMAT laboratory to perform mechanical tests at elevated temperatures under controlled atmosphere on micrometer-thick specimens [30]. The investigated temperatures were 650, 700, 750, 800 and 850 $^\circ\text{C}$. A special attention was paid to the atmosphere (1.2 bar high purity Ar static atmosphere combined with oxygen getter made of particles of Zr) to prevent the high surface/volume-ratio specimen from surface reactivity. Surface degradation (e.g. oxidation,

sublimation) might considerably affect the mechanical properties of the material investigated. Dimensional variations of the specimens due to either the mechanical loading or the temperature variation were continuously measured with a Keyence LS-7030M optical micrometer. The sample was heated with an unfocused 2-zone halogen lamp furnace to ensure a low thermal gradient along the gage zone ($< 2 \text{ }^\circ\text{C}$). The temperature was measured at two locations with very thin S-type thermocouples (76 μm diameter) spot-welded on platinum plates (90 μm thickness), located close to the gage zone. For the tensile experiments, tests were conducted under controlled crosshead displacement to impose a strain rate of $5 \times 10^{-5} \text{ s}^{-1}$ at temperature.

3. Results

3.1. Microstructure of the MCrAlY Tribomet® coating

Following the standard diffusion heat treatments, the “as-received” microstructure of the NiCoCrAlYTa coating was β - γ - γ' (Fig. 2). This microstructure consisted in $\text{B2-}\beta$ -(Ni,Co)Al and L1_2 - γ' -(Ni,Co,Cr) $_3$ (Al,Ti)

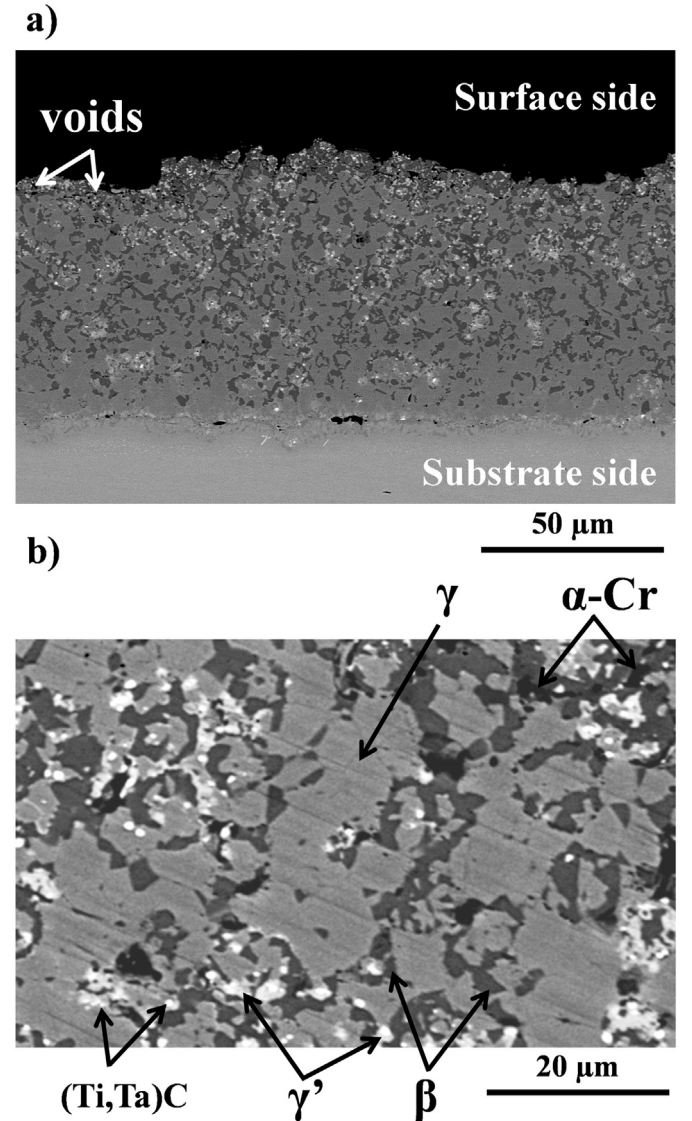


Fig. 2. “As-received” microstructure of the NiCoCrAlYTa coating illustrating the morphology and the repartition of the β , γ , γ' phases. a) Cross-section observation of the 70 μm -thick coated Ni-based superalloy. A high fraction of small voids were noticed in the near surface region of the coating, b) Morphology and spatial distribution of the different phases reminding the spherical pattern of the original CrAlYTa particles. Additional phases such as $(\text{Ti,Ta})\text{C}$ and α -Cr precipitates were documented.

phases entrapped in a DO_{22} - γ -Ni matrix, depicted as dark, light and intermediate grey in Fig. 2.b, respectively. Interestingly, β and γ' phases formed rounded patterns with a diameter in a range between 5 and 15 μm , corresponding to the original CrAlYTa particles geometry and size used in the Tribomet® deposition process. Additional precipitates sparsely dispersed within the coating were found such as (Ta,Ti)C (white precipitates in Fig. 2.b) and α -Cr (dark grey precipitates, darker than the β phase in Fig. 2.b), this latter phase being hardly distinguishable from the β phase in an electron backscattered mode. EDX mappings aimed to better illustrate the chemical element partitioning between the different phases. The brittle α -Cr phase, rich in chromium and depleted in nickel (as depicted in Fig. 3.d & b, respectively), was clearly highlighted with element distribution maps. This phase was mostly

located in a shallow region beneath the surface, in the vicinity of β and γ' phases. A high fraction of voids was also noticed in this shallow region beneath the surface (Fig. 2.a). (Ta,Ti)C precipitates were found through the whole section of the coating (see e.g. Ta distribution in Fig. 3.g), dispersed on spherical patterns reminding the original CrAlYTa particles. Thin and thick coatings had a similar microstructure, as expected for overlay coatings.

As illustrated in Fig. 2.a, MCrAlY Tribomet® coatings demonstrated a relatively high roughness since the maximum height of profile roughness R_z is nearly 15 μm (tail of the CrAlYTa particle distribution size). Therefore, 10 μm were polished from the top surface of the coating in order to substantially reduce the roughness, leading to flat polished regions and sparse valleys (Fig. 4.a). Cross-section analyses of the sparse

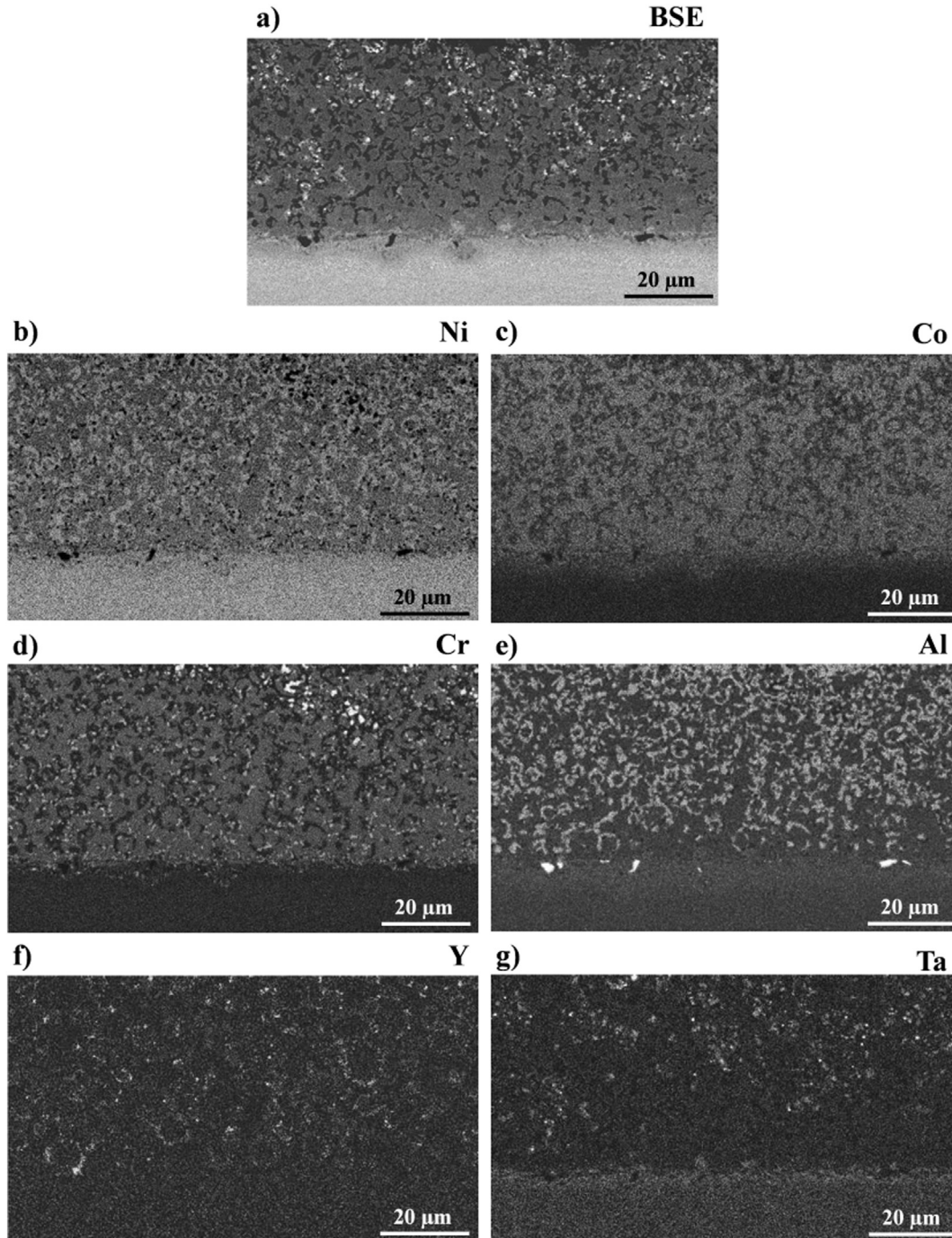


Fig. 3. Microstructure and element maps obtained via EDX analyses of the NiCoCrAlYTa Tribomet® coating: a) Cross-section observation of the coating in a backscattered electron mode. Element maps showing element partition between the different phases b) Ni, c) Co, d) Cr, e) Al, f) Y and g) Ta.

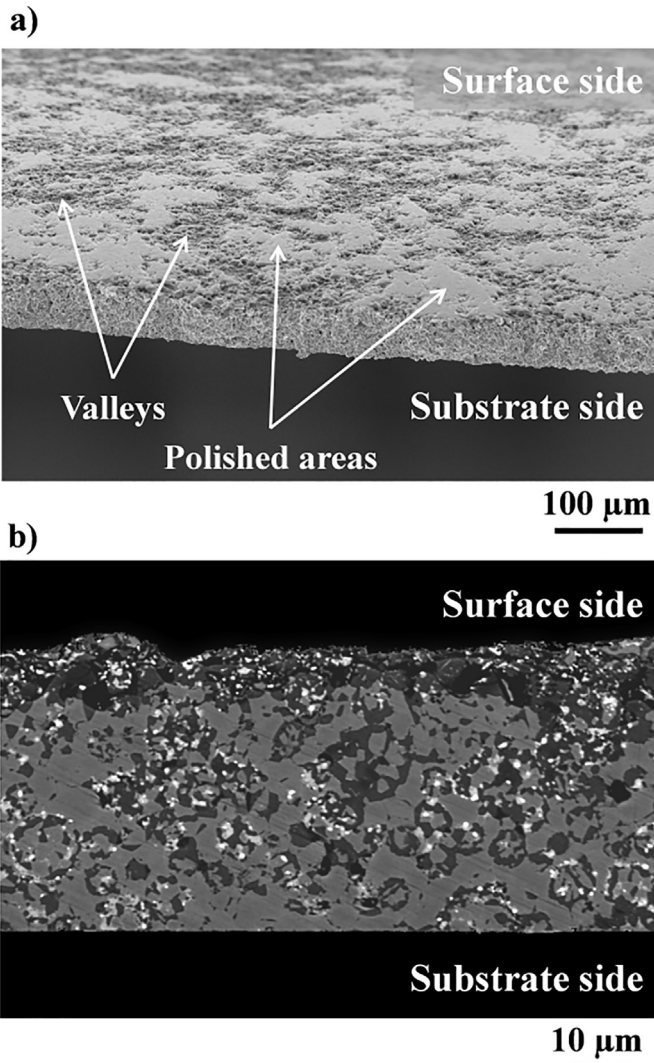


Fig. 4. a) Surface integrity of the polished NiCoCrAlYTa Tribomet® coating showing polished areas and valleys. b) Cross section of a thin freestanding coating specimen depicting the β -phase rich outer layer of the coating.

valleys aimed to evaluate the effective cross-section of thin FSCS required for further mechanical strength characterization. Cross-section observations of the coating also evidenced a high surface fraction of the β phase in a shallow region beneath the polished area and valleys surface, leading to a thin roughly continuous β phase layer with some embedded α -Cr phases and small voids at the extreme surface of the coating (Fig. 2.a and 4.b). The as-received MCrAlY Tribomet® microstructure differed from the outer side and the inner side of the coating, potentially due to the deposition process, the interdiffusion with the substrate and/or environmental effects during standard heat treatments.

3.2. Tensile properties of the NiCoCrAlYTa coating

3.2.1. Elastic properties of the bulky NiCoCrAlYTa FSCS

The dynamic elastic properties of the bulky NiCoCrAlYTa FSCS were assessed using the dynamic resonant frequency method in a bending vibration mode at various temperatures ranging between 20 and 1000 °C (Fig. 5). The dynamic elastic modulus of the coating was found to be 184 GPa at room temperature and slightly decrease to 150 GPa with the temperature increase up to 700 °C. The dynamic elastic modulus was shown to linearly decrease with the temperature between 20 °C and 600 °C. At temperatures above 700 °C, the dynamic elastic

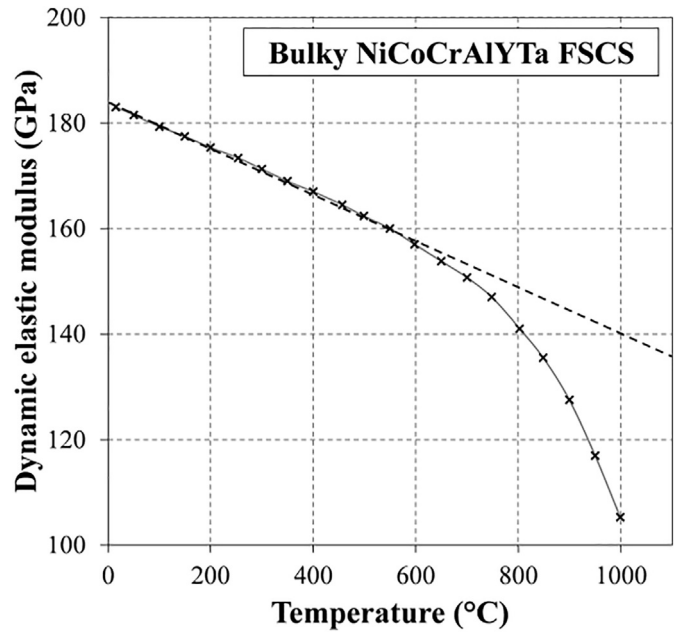


Fig. 5. Evolution of the dynamic elastic modulus of the bulky NiCoCrAlYTa Tribomet® coating between 20 and 1000 °C.

properties of the coating rapidly dropped off to reach a value of 105 GPa at 1000 °C.

3.2.2. Tensile properties on thin NiCoCrAlYTa FSCS

Tensile tests on thin NiCoCrAlYTa FSCS were performed between 650 °C and 850 °C. As illustrated in Fig. 6, the mechanical strength (0.2 pct offset yield stress (Y.S.) and ultimate tensile strength (U.T.S.)) of the thin FSCS progressively decreased with the temperature increase while the ductility considerably increased. At 650 °C, the coating fails in a brittle manner and withstands 500 MPa before failure. At 700 °C, necking of the sample is noticed on the stress-strain curve showing a starting of ductility. Above 800 °C, the strain-to-failure of the coating exceeded 5% ($S-T-F_{\text{thin FSCS}}(800\text{ °C}) = 5.5\%$ and $S-T-F_{\text{thin FSCS}}(850\text{ °C}) = 6.2\%$). At 850 °C, the yield strength and the ultimate tensile strength of the thin FSCS were 58 MPa and 65 MPa, respectively. It is worth noting that the effective cross-section of the thin FSCS was used

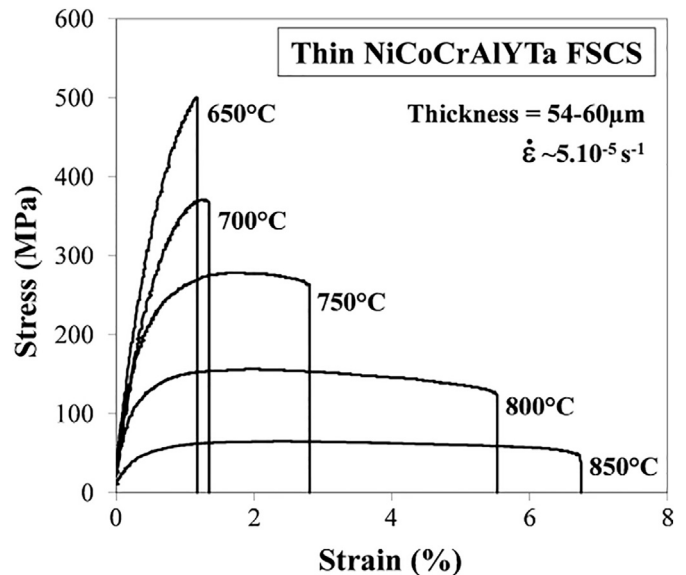


Fig. 6. Stress-strain curves of the thin NiCoCrAlYTa Tribomet® FSCS between 650 °C and 850 °C.

for the calculation of the mechanical strength, i.e. the gage section of the thin FSCS reduced by the average surface of valleys measured on several cross-sections (Fig. 4.a).

The tensile properties (Y.S., U.T.S. and strain-to-failure) obtained on the thin NiCoCrAlYTa FSCS were compared with the ones of the bulky FSCS (Fig. 7 and Fig. 8). For both the bulky and thin FSCS, Y.S. and U.T.S. linearly decreased when the temperature increased from 650 °C to 850 °C. Similar trends and comparable values were found for both the coating thicknesses in terms of mechanical strength; thin FSCS exhibited yet slightly lower mechanical strength than bulky FSCS. As far as the ductility of the coatings is concerned, the strain-to-failure for thin and thick FSCS was comparable and increased with the temperature. Thin FSCS were shown to have a lower ductility than bulky FSCS at temperatures close to the BDTT. Inversely, the ductility of thin FSCS is higher at higher temperatures. Additional room temperature tensile tests were performed on bulky NiCoCrAlYTa FSCS. Bulky FSCS demonstrated high mechanical strength at room temperature, i.e. $Y.S._{bulky FSCS}(20\text{ }^\circ\text{C}) = 875\text{ MPa}$ and $U.T.S._{bulky FSCS}(20\text{ }^\circ\text{C}) = 980\text{ MPa}$, which are about 50% higher than those at 650 °C.

3.3. Fracture area of thin NiCoCrAlYTa FSCS

Fractographic analyses were conducted on the thin FSCS tested at different temperatures (Fig. 9). First of all, it is worth mentioning that the fracture surface was not homogeneous whatever the tested temperature. Indeed, an outer layer - denoted "surface side" in Fig. 9 and nearly as thick as a CrAlYTa particle diameter - was clearly distinguishable from the remaining fracture surface. In this region, spheroidal and non-cohesive particles were found both in the valley and polished areas (areas defined in Fig. 4.a). This outer layer was thicker in valley areas than polished areas. This observation can easily find justification in the fact that this outer layer follows the initial topography/rough surface of the as-deposited coating. In the valley areas, several voids were found between the spherical particles (Fig. 9). In the remaining region of fracture surface, spherical features also appeared for coatings tested up to 850 °C but in a different manner compared to those found in the outer layer. These spherical features are typical of a cohesive but brittle intergranular/interparticulate fracture. High magnification observations on those spherical features did not reveal strong differences in terms of fracture mechanisms even 200 °C above the BDTT (Fig. 10.a to c). The multiphase microstructure of the coating, especially in those intergranular/interparticulate regions, makes the fracture surface difficult to interpret. However, sharper features at the intergranular/interparticulate fracture were noticed for lower temperatures. Furthermore, one tensile test was tried to be conducted at 950 °C but the thin FSCS failed during

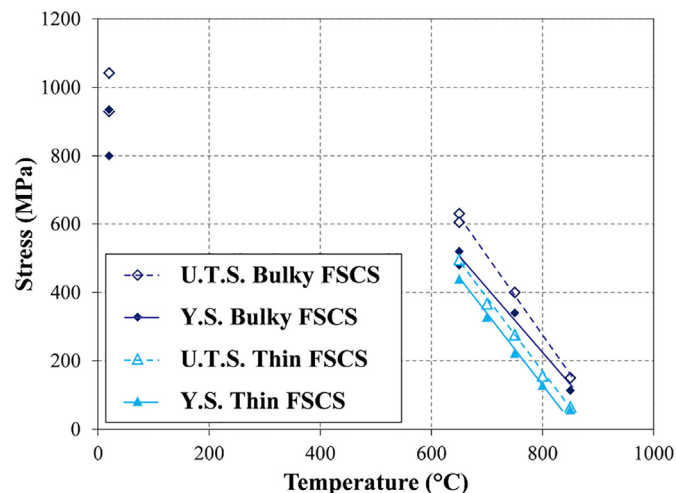


Fig. 7. Evolution of the mechanical strength (yield stress (Y.S.) and ultimate tensile strength (U.T.S.)) with the temperature for thin and bulky NiCoCrAlYTa Tribomet® FSCS.

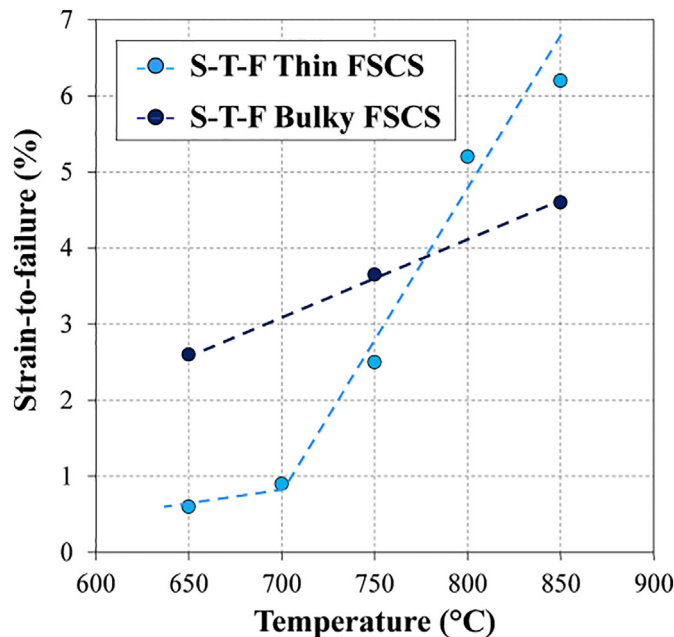


Fig. 8. Evolution of the strain-to-failure (S-T-F) with the temperature for thin and bulky NiCoCrAlYTa Tribomet® FSCS.

the 950 °C-dwell prior to the tensile test. The fracture surface of the 950 °C thin FSCS (see Fig. 10.d) depicted fracture features substantially different from thin FSCS tested below 850 °C (Fig. 10.a to c). For temperatures as high as 950 °C, the smoother and less particular fracture surface strongly vouched for the high ductility of the coating. Fracture surfaces also highlight the occurrence of voids inside the coating.

4. Discussion

The tensile properties of a β - γ - γ' MCrAlY coating obtained with a non-line-of-sight deposition process were investigated in a wide range of temperatures. The main goal of the present paper was to examine both the mechanical strengths and the deformation capabilities of such a processed coating in relation with the temperature. The high temperature mechanical characterization of an overlay MCrAlY coating, processed with two different thicknesses with the same chemical composition and microstructure, were performed. The thin coating materials (70 μm), representative of coatings used for aeronautical turbine blade applications, were compared with bulky coatings (350 μm), a "reference material" capable to be tested with conventional mechanical test rigs. The comparison between the two-thickness materials aimed to validate a recent test rig developed for the mechanical characterization of thin freestanding specimens at elevated temperature under a specific controlled atmosphere. Since very comparable values between those two specimen geometries, macroscopic mechanical properties (elastic dynamic modulus, yield stress, ultimate tensile strength, and strain-to-fracture) are thus possible to be assessed via such a microscopic specimen investigation. The relatively long dimensions of the specimens (centimeter-long and millimeter-wide gage) versus the specimen thickness (tens of micrometers) are sufficient to sample a representative elementary volume of the material.

4.1. Mechanical behavior of the NiCoCrAlYTa coating at high temperature

As far as the tensile behavior of the β - γ - γ' MCrAlY coating is concerned, the dynamic elastic modulus and the mechanical strength of the coating decreased for temperatures above 650-700 °C while the ductility substantially increased. Therefore, the BDTT of this specific coating was found to lie between 650 and 700 °C. Fig. 11 summarizes

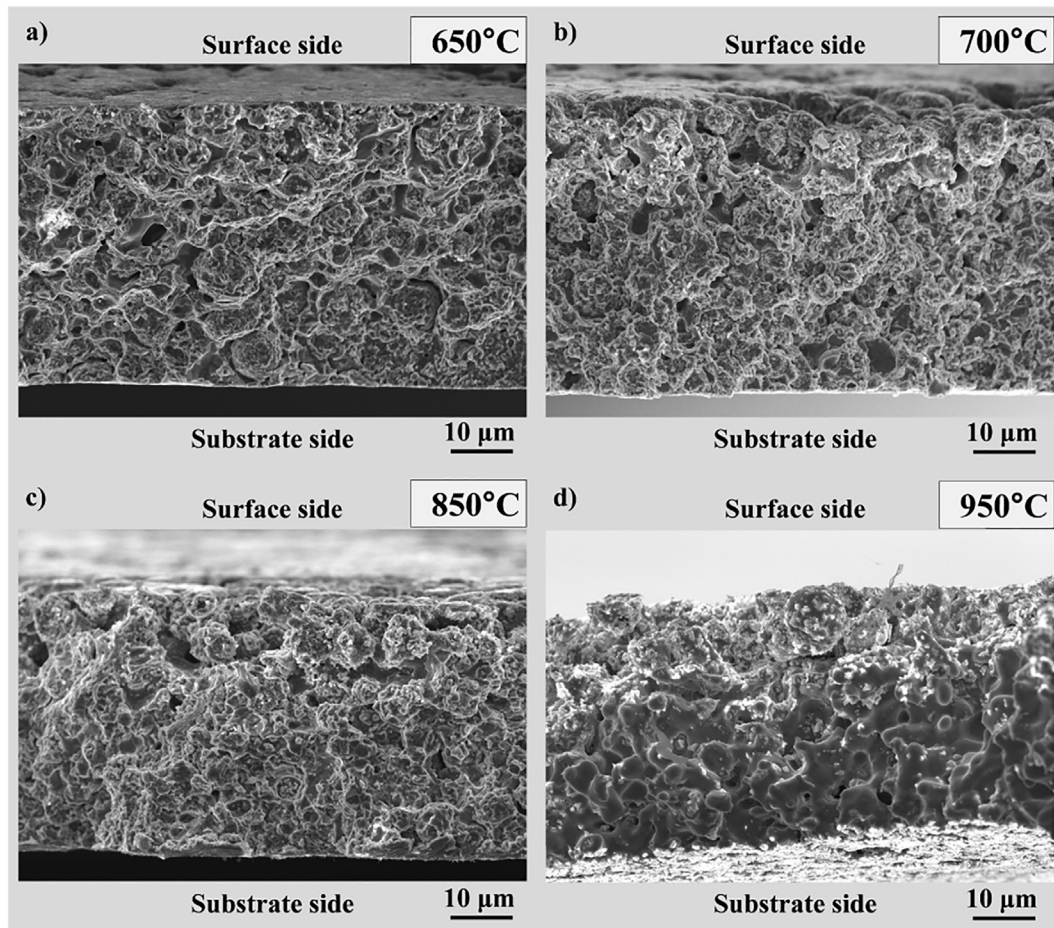


Fig. 9. Fracture surface of the thin NiCoCrAlYTa FSCS tested at different temperatures. a) 650 °C, b) 700 °C, c) 850 °C, d) 950 °C.

the evolution of the different mechanical properties measured in the present study normalized by the interpolated property at 675 °C. While the mechanical strength (yield stress and ultimate tensile strength) and ductility are properties very sensitive to the temperature in the vicinity of the BDTT, the evaluation of the dynamic elastic properties presents the advantage to identify this in-temperature transition behavior in a non-destructive manner. This method might be applicable to coated substrate if the substrate is not subjected to properties variation in the range of temperatures.

4.2. Brittle-to-ductile transition temperature and mechanical properties: comparison with other coatings

The literature on the BDTT of MCrAlY coatings is limited and values reported are comprised in a large range of temperatures ($BDTT_{overlay\ coat.} = 235\text{--}910\text{ °C}$ [16,17]). This large range of BDTT is mainly due to the variety in chemistry and microstructure possible with overlay coatings. Indeed, the occurrence and fraction of high-BDTT phases, such as β -NiAl [3,4,19–23] and α -Cr phases [34], tend to increase the BDTT [3, 4,16,17]. Based on the experience of aluminized coatings, a decrease in aluminum content leads to a decrease of BDTT due to the higher fraction of γ -Ni phase, and inversely the lesser fraction of γ' -Ni₃Al and β -NiAl phases. Depending on the stoichiometry of the β -NiAl phase, its BDTT ranges from 600 to 970 °C [3,4,19–23] while the one of the γ -Ni phase is below 0 °C [3]. According to the non-negligible fraction and the interconnected morphology of β -NiAl phase in the Tribomet® NiCoCrAlYTa coating, the intermediate BDTT found in the present investigation is consistent with the literature. Interestingly, NiCoCrAlYTa coatings obtained with the Tribomet® process exhibit an intermediate mechanical strength compared to Pt-modified aluminized coatings but a

significantly higher ductility [19,20] and considerably higher mechanical strengths than β - γ - γ' NiCoCrAlY coatings [35]. Indeed, the strain-to-failure of the thin NiCoCrAlYTa FSCS ($S\text{-}T\text{-}F_{Tribomet}(BDTT + 200\text{ °C}) \approx 6.2\%$) above the BDTT was significantly higher than the one noticed for Pt-modified aluminized FSCS ($S\text{-}T\text{-}F_{NiAlPt}(BDTT + 200\text{ °C}) \approx 2.0\%$ [19,20]).

4.3. Fracture surface: deformation and fracture mechanism

While the coating experiences significant ductility above the BDTT ($S\text{-}T\text{-}F(850\text{ °C}) = 6.2\%$), fracture surface of specimen tested at this temperature (Fig. 9.b&c and Fig. 10.c&d) appears slightly brittle and very comparable to the one observed at lower temperatures, below BDTT (Fig. 9.a and Fig. 10.a&b). Intergranular/interparticular fractures were noticed without particular tearing of the γ -Ni phase, as reported by Chen et al. on a β - γ CoNiCrAlY coating [29]. In the present study, the brittle β -phase is decorating the original CrAlYTa particles envelop while the ductile γ -phase constitutes the inner region of the CrAlYTa particles and the surrounding matrix. While the ductility is significantly improved due to the intragranular/intraparticular deformation, the fracture mechanism remains slightly brittle due to the interconnected β -phase still brittle at this temperature. The strong cohesion between particles is conferred by the diffusion heat treatments (1080 °C–6 h + air cooling followed by 820 °C–20 h + air cooling). When increasing the testing temperature above 900 °C (over the BDTT of the β -phase), the fracture surface of the NiCoCrAlYTa coating is considerably different and typical of a highly ductile fracture. From an application point of view, some improvements in the heat-treatment process could be considered in order to reduce the interconnected arrangement of the high

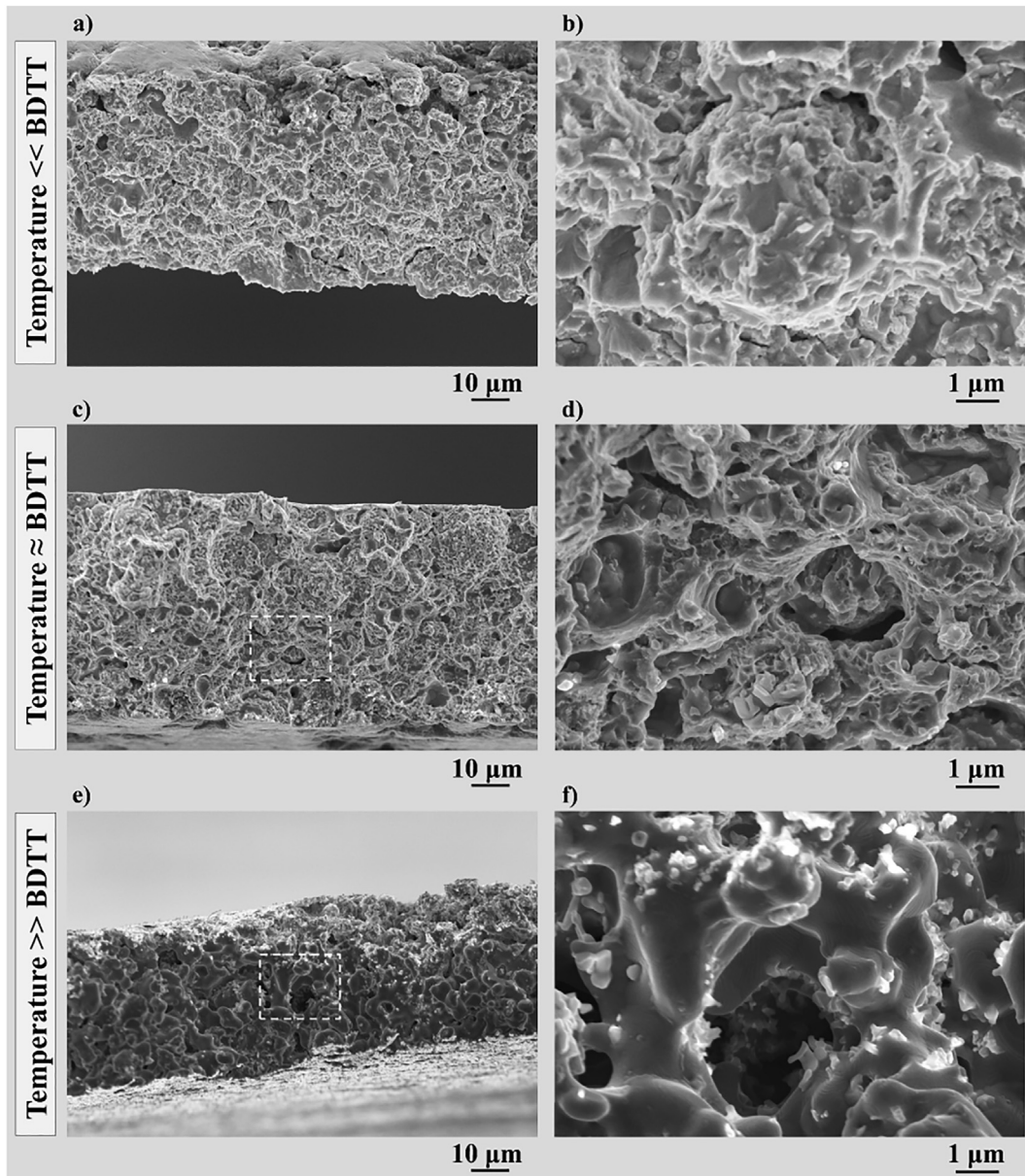


Fig. 10. Low and high magnification observations of the fracture surface of the NiCoCrAlYTa coatings at various temperatures: a & b) below the brittle-to-ductile transition temperature (BDTT, i.e. $T \leq 650$ °C); c & d) at close to the BDTT ($T \approx 650$ °C–850 °C); e & f) above the BDTT ($T \geq 950$ °C).

temperature brittle phase and to subsequently gain in ductility (β -phase).

4.4. Surface integrity due to the Tribomet® deposition process

Fracture surface analyses aimed also to identify a different fracture mechanism in the extreme surface of the coating, less cohesive and more brittle. This difference in local mechanical behavior was shown to correspond to a β -NiAl and α -Cr rich layer in the extreme surface of coating, inherent to the deposition process. The roughness associated with this process and the high void fraction in this outer layer (Fig. 2.a) might also contribute to the loss of mechanical properties between the two geometries of tested specimens. The combination of high fraction of brittle phases and high porosity in this process affected region (≈ 10 μm , i.e. ≈ 16 –18% of the thin FSCS thickness) is strongly believed to participate in the slightly lower mechanical strength results found for thin FSCS compared to bulky FSCS (exempt of surface defect). Despite those negative aspects, the NiCoCrAlYTa Tribomet® coating exhibits impressive mechanical

properties, especially for a non-line of sight deposition process. Additional plating on this coating is possible to lower the roughness of the coating, better match dimensional tolerances (thickness control and uniformity for complex geometry) [31] and might improve the cohesion of the outer layer.

5. Conclusions

The tensile properties of a NiCoCrAlYTa coating obtained by the non-line of sight Tribomet® process were characterized in a large range of temperatures using freestanding coating specimens (FSCS). Mechanical characterizations, fractographic analyses and cross-section observations aimed to identify different conclusions:

- 1- The brittle-to-ductile transition temperature (BDTT) of this β - γ - γ' MCrAlY coating was found between 650 °C and 700 °C. Above this temperature, the coating exhibited a lower mechanical strength (yield stress and ultimate tensile strength), a loss of elastic properties and a higher ductility.

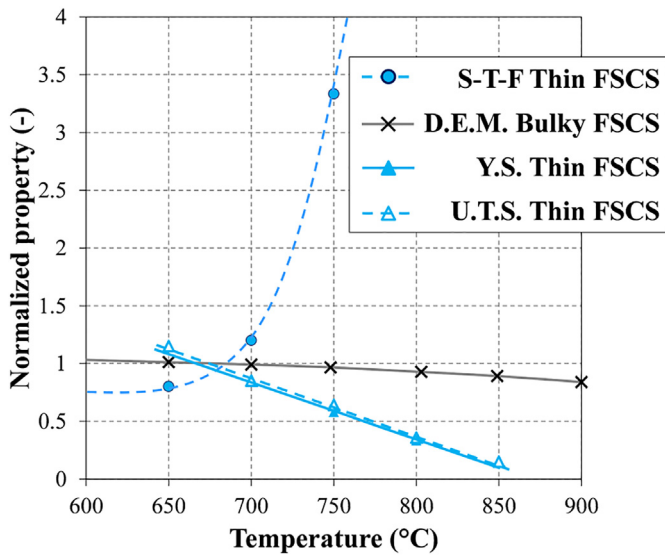


Fig. 11. Evolution of the different mechanical properties of the thin and bulky NiCoCrAlYTa FSCS as a function of the temperature. The properties are normalized by the average properties at 650 °C–700 °C.

- Fracture surface analyses highlighted kind of brittle intergranular/interparticular fracture even above the BDTT with macroscopic deformation as large as 6.2%. This observation find explanation in the fact that the deformation was mainly intragranular/intraparticular while the fracture mechanism remained brittle due to the well-distributed network of the β -phase, still brittle at 750–800 °C.
- Consistent results were found between conventional bulky and thin FSCS. It demonstrates the feasibility of such micromechanical characterizations at high temperature.
- A less cohesive and more brittle region was found in the outer layer of the coating due to the deposition process. This region corresponds to a β -NiAl and α -Cr rich layer that might be avoided by tribofinishing techniques or by additional plating. This process-affected region results in a slightly lower mechanical strength (Y.S. and U.T.S) of thin NiCoCrAlYTa FSCS.
- The NiCoCrAlYTa coating demonstrated interesting mechanical strength, impressive ductility for freestanding specimens and a relatively low BDTT. The combination of all these properties makes this coating an interesting candidate for coated structural components and justifies its intensive use for severe high temperature applications.

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