



**HAL**  
open science

# Mesure et Evolution des gradients de propriétés mécaniques dans le système superalliage à base de nickel MC2 revêtus MCrAlY

Damien Texier, Daniel Monceau, Eric Andrieu

► **To cite this version:**

Damien Texier, Daniel Monceau, Eric Andrieu. Mesure et Evolution des gradients de propriétés mécaniques dans le système superalliage à base de nickel MC2 revêtus MCrAlY. CFM 2013 - 21ème Congrès Français de Mécanique, Aug 2013, Bordeaux, France. hal-03439737

**HAL Id: hal-03439737**

**<https://hal.science/hal-03439737>**

Submitted on 22 Nov 2021

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

# Measure of the gradient of mechanical properties in MCrAlY coated MC2 Ni-base superalloy system at high temperature

D. TEXIER<sup>a</sup>, D. MONCEAU<sup>a</sup>, E. ANDRIEU<sup>a</sup>,

a. CIRIMAT (UMR 5085), 4 Allée Emile Monso, 31030 TOULOUSE

## Résumé :

*Le gradient de propriétés mécaniques existant dans un système superalliage revêtu a été quantifié dans le domaine de température représentatif des conditions de fonctionnement d'une aube de turbine. Pour se faire, des échantillons ultraminces (20-100µm) ont été prélevés dans les différentes zones de ce gradient de microstructure (le revêtement, la zone d'interdiffusion et le superalliage). Ces échantillons ont ensuite été testés sur des bancs d'essais dédiés aux échantillons d'épaisseurs micrométriques. Les propriétés mécaniques mesurées sur éprouvettes de type zone d'interdiffusion montrent bien le caractère non porteur de cette zone lorsque le système est sollicité en fluage à très haute température.*

## Abstract :

*Gradient of mechanical properties existing in coated superalloys system has been quantified in the temperature range corresponding to turbine blades operating conditions. Hence, ultrathin tensile specimens (20 to 100µm thick samples) were extracted in the different zones of the gradient of microstructure i.e (coating, superalloy substrate and interdiffusion zone). Specimens were then tested on mechanical test rigs dedicated to specimens with micrometric thicknesses. Results concerning the mechanical behavior of the interdiffusion zone led to conclude that this zone of the microstructural gradient had to be considered as a non bearing zone in creep conditions representative of service operating conditions.*

**Mots clefs :** superalliage, revêtement MCrAlY, interdiffusion, échantillons ultraminces, traction

**Keywords :** superalloy, MCrAlY coating, interdiffusion, ultrathin specimen, tensile experiments

## 1 Introduction

Ni-based superalloys designed for single crystal gas turbine blades undergo severe service conditions such as mechanical, thermal and environmental solicitations. The chemical composition and also the microstructure of these superalloys have been optimized through different generations to resist creep deformation at temperature higher than 1100°C [1, 2]. To protect these materials from environment stresses (isothermal and cyclic oxidation at high temperature, intermediate temperature corrosion) blades are generally coated [3]. However, both coating process and associated thermal treatment favor the interdiffusion of different chemical elements between the coating and the superalloy. This phenomenon generates an interdiffusion zone (I.Z.) between the microstructures of the coating and the substrate. The microstructure of the different strata (superalloy (S.Z.), coating (C.Z.) and interdiffusion zone (I.Z.)) evolves in service conditions due to the combined effect of high temperature and stress. The variation of the chemical composition of the system in the different zones directly modifies the coefficient of thermal expansion [4], the mechanical properties [5, 6] or even the lattice parameter of the phases constitutive of the system. Refinement of predictive models on the mechanical behavior and lifetime of materials needs the characterization of the local properties of each zone to be carried out, especially on both sides of the original interface. To characterize these functionally graded materials at the scale of the microstructural evolutions, different technical means exist for the study of mechanical and physical properties at room temperature. Nevertheless, regarding the investigation of the high temperature properties [5-11], other tools have to be developed. The aim of our study was to characterize at a local scale the tensile and creep properties of the different zones constituting the system (as received and aged) as well as their thermal expansion. This database was not available up to now.

## 2 Material

The system investigated in this study is a first generation single-crystal nickel based superalloy (MC2) coated with a 70 $\mu\text{m}$  thick NiCoCrAlYTa layer, elaborated by electrochemical deposition (TRIBOMET Process). The gradient of microstructure is illustrated in FIG-1. It has been divided into three zones: the monocrystalline substrate (S.Z.), the coated zone (C.Z.), and the interdiffusion zone (I.Z.). The microstructure corresponding to each zone is detailed below.

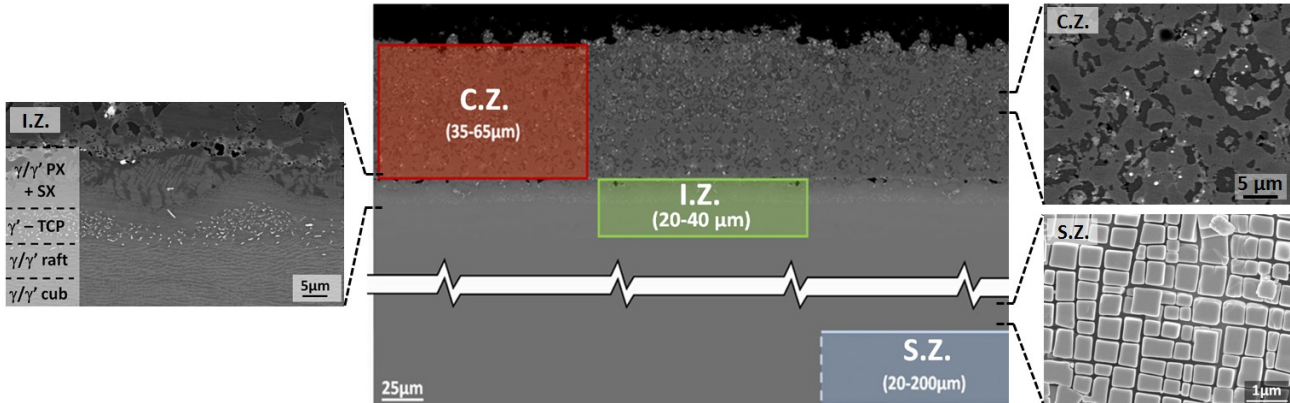


FIG- 1 : Global view of the gradient of microstructure existing in a NiCoCrAlYTa coated MC2 (BSE-SEM). Magnification of the investigated zones (C.Z. (BSE-SEM), I.Z. (BSE-SEM), S.Z. (SE-SEM))

### 2.1 The substrate : MC2 (S.Z.)

The nominal composition of this first-generation single crystal superalloy has been measured with EPMA analyses (TAB-1). Microstructure of the « as received » superalloy consists in a regular arrangement of  $\gamma'$ -L1<sub>2</sub> precipitates (light grey), aligned along the  $\langle 001 \rangle$  directions of the  $\gamma$  fcc single crystal solid solution matrix (dark grey). This microstructure, optimized for high-temperature creep performance, is the result of standard heat treatments (1300°C-3 h + air cooling (A.C.) – 1080°C-6 h + A.C. – 870°C-20 h + A.C.).

### 2.2 The Coating : NiCoCrAlYTa (C.Z.)

The mean chemical composition of the coating is given in TAB-1. Spherical motifs corresponding to the CrAlYTa particles used during the deposition process appear on the micrograph.  $\beta$ -(Ni,Co)Al phases (dark grey) are dispersed into a  $\gamma$  matrix (intermediate grey), containing  $\gamma'$ -(Ni,Co,Cr)<sub>3</sub>(Al,Ti,Ta) phases (light grey). Dispersion of precipitates rich in titanium and tantalum (Ta,Ti)C (white precipitates) is relatively homogeneous along the thickness of the coating.

### 2.3 The interdiffusion zone (I.Z.)

The stratified microstructure, characteristic of the interdiffusion zone, is the result of the cross diffusion of chemical elements between the coating and the substrate, and also the result of parameters from the deposition process. The interdiffusion zone corresponds to the thickness of superalloy affected by the presence of the coating. This one is delimited by different markers: alumina coming from sand-blasting (black particles) located at the initial position of the substrate-coating interface and (Ta,Ti)C precipitates (white particles). A  $\gamma/\gamma'$  “palm tree” looking like microstructure appears below. It comes from the cellular recrystallisation due to the mechanical treatment before deposition (sand-blasting). This area grows during the thermal treatment of diffusion (R1) at 1080°C [12]. Then, a singlecrystalline  $\gamma/\gamma'$  strata comes, delimitating both the zone of cellular recrystallisation and the  $\gamma'$  layer peppered with TCP phases. Finally, a  $\gamma/\gamma'$  microstructure rafted parallel to the coating-substrate interface can be observed. This last zone seems to demonstrate the effect of sand-blasting too, generating a bi-axial compressive state in the substrate before the deposition of the coating [Bie' 00]. This type P rafting is in accordance with the negative misfit existing between  $\gamma$  and  $\gamma'$  phases at 1080°C. The R1 heat treatment is responsible of the rafting of the  $\gamma/\gamma'$  microstructure.

TAB- 1: Chemical composition of the superalloy and the coating (% at.)

<i>Eléments</i>	<i>Ni</i>	<i>Cr</i>	<i>Co</i>	<i>Mo</i>	<i>W</i>	<i>Al</i>	<i>Ti</i>	<i>Ta</i>	<i>Y</i>
$X_{MC2}$ (EPMA)	Base	8,93	5,07	1,27	2,48	10,67	1,83	2,00	-
$X_{NiCoCrAlYTa}$ (EDX)	Base	19,81	17,23	0,17	0,47	17,19	0,1	1,28	0,79

### 3 Experimental procedures

In order to characterize the mechanical properties of these different zones from room temperature to very high temperature, ultrathin specimens have been extracted from this system. The thickness mentioned on the global view (figure-1) correspond to the specimens thicknesses used in this study. It is then necessary to test them mechanically at high temperature with dedicated test rigs developed in the laboratory.

#### 3.1 Extraction of the specimens

To reach the objective mentioned above, an extraction method of material into these micronics thick zones has been set up as a part of this work. The targeted thicknesses were defined in order to fit with the thicknesses of the different microstructures of the system. These specimens, whose thicknesses are in a range from 20 to 200 $\mu$ m, were prepared using a LOGITECH CL50 lapping machine associated with a PP5GT precision Jig in order to limit the impact of grinding on the residual stresses. Optimization of this method (position and geometry of the specimens) provides a variation in thickness of the gauge length close to 1.5  $\mu$ m maximum. In our case, the gauge length is between 30 to 40mm long and 2mm wide. This effort in development enables to use specimens with conventional sizes comparatively to other studies using so thin specimens, but in a “dog bone” shape [13]. This “dog bone” geometry is under controversy when dealing with the determination of flow rules. Figure-2 highlights the thicknesses obtained in this study through the illustration of the cross sections of a C.Z. and a I.Z. specimen.

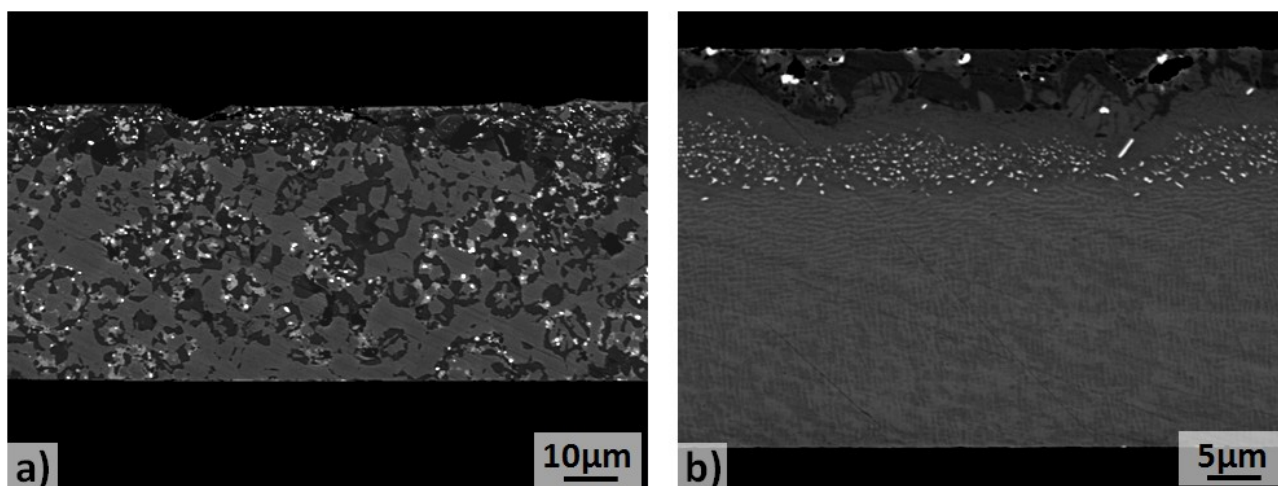


FIG-2: Cross sections of two specimens: a) C.Z. type, b) S.Z. type

#### 3.2 Mechanical test rigs

Two mechanical test rigs have been especially designed in this study to carry out tensile and creep experiments on ultrathin specimens. These tests have been conducted under controlled atmosphere up to 1200°C. Environmental conditions have been optimized in order to limit the effects induced by oxidation on the mechanical behavior of these ultrathin specimens (depleted zone, vacancy injection). Mechanical tests were conducted under a 1.2 bar high purity Ar static atmosphere combined with O<sub>2</sub> getter in Zr in order to reduce the partial pressure of oxygen into the vessel. Care has been also taken with water desorption. One of the test rigs is equipped with an actuator in order to make the load evolve in time. The second one is a dead-weight application – the applied load is constant during a creep experiment. Experiments can be conducted in a loading range from 2 to 1000N. On account of the thicknesses of the specimens, it was not conceivable to use a contact method for measuring as well lengthening as temperature. Therefore, the strain measurement

was performed with an optical micrometer. Temperature measurement was carried out at two locations with S thermocouples welded on platinum plates, located close to the gauge length. In this study, tensile experiments were conducted under a strain controlled rate equal to  $10^{-4}s^{-1}$ .

### 4 Results

Figure-3 illustrates results from tensile tests carried out on the coating of the specimens between 650°C and 800°C. The mechanical behavior of the coating (yield stress and ultimate tensile strength) decreases with the temperature between 650°C and 800°C. The quasi-brittle behavior of the coating was evidenced at 650°C. The ductility of the coating increases with temperature. In that case, the brittle to ductile temperature transition (BDTT) is close to 650°C.

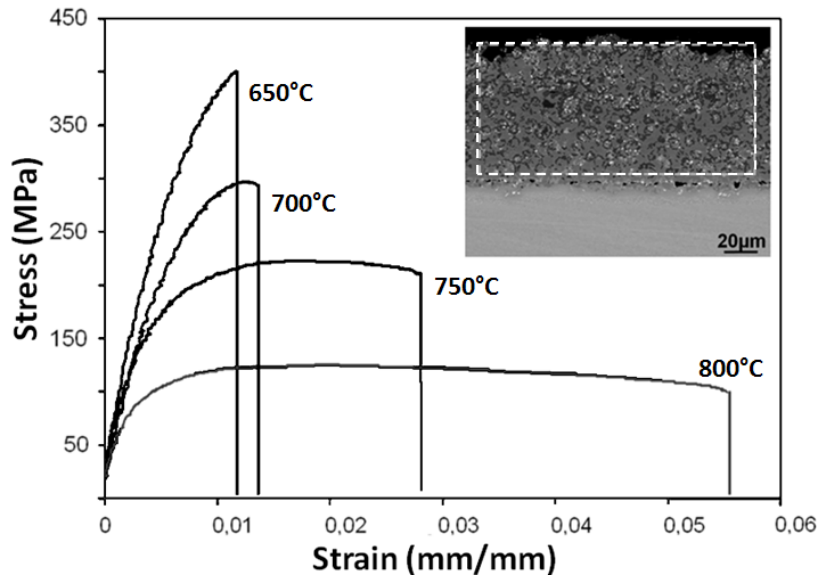


FIG-3: Results of tensile experiments carried out on C.Z. specimens

Extracting specimens and performing such mechanical tests enable to characterize the intrinsic behaviour of the interdiffusion zone and the substrate. The results from these experiments highlight the gradient of mechanical properties existing between the inner and outer layers constituting the MC2/NiCoCrAlYTa system. A synthesis of these results is presented in figure-4. In service conditions, the mechanical behaviour of the superalloy is clearly higher than the one corresponding to interdiffusion zone. Because of poor mechanical resistance of the coating in this range of temperature, intermediate temperatures were investigated.

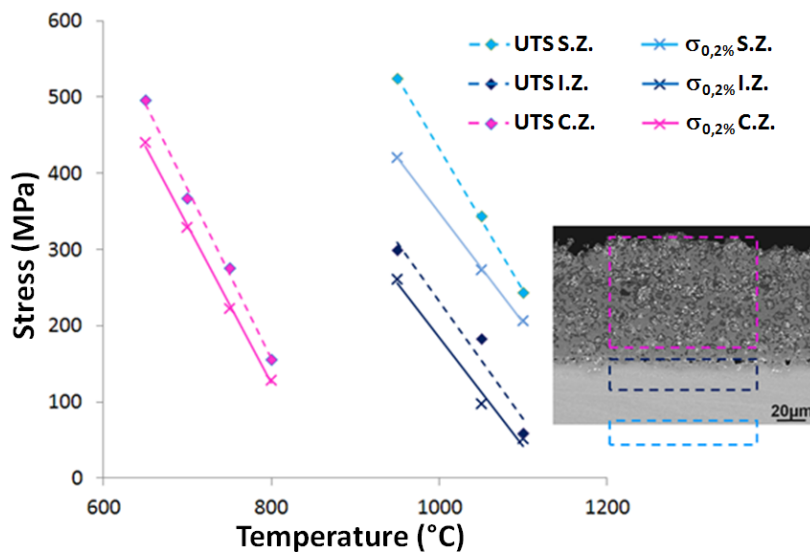


FIG-4: Synthesis of mechanical properties illustrating the gradient of mechanical properties (yield stress:  $\sigma_{0,2\%}$  and ultimate tensile strength: UTS)

## 5 Discussion

Despite the coherency of the results obtained on ultrathin specimens, scientific issues can be dressed up: Is it possible to characterize volume properties of a material at room temperature as well as at high temperature from ultrathin specimens? Thinner the thickness of the specimen is, the more important the Surface/Volume ratio is. Therefore, the challenge is there to avoid surface damages (high temperature oxidation, sublimation, nitridation) and to avoid “size effects” from a mechanical point of view.

To answer these questions, we compared the results from ultrathin specimens with the database existing on bulk materials. Both the coating and the superalloy have already been tested in the same range of temperature. That’s why, the mechanical characterization of these materials does not constitute the innovative part of this work but a benchmark regarding to the validity of our experimental tools. The innovation of this work is the characterization of mechanical properties of materials extracted from systems at industrial scales, and especially of the interdiffusion zone which can’t be elaborated as bulk material. For instance, Boudot elaborated hundreds of micrometer thick layer of NiCoCrAlYTa and carried out mechanical tests on conventional test rig [14]. The determination of static mechanical constants (yield stress and ultimate tensile strength) is different from 5% between both studies. Furthermore, we noticed less than 10% in difference in mechanical behavior between ultrathin specimens and bulk materials in MC2. As far as the surface effect is concerned, we previously carried out 40h long creep tests in the same atmosphere. Surface analyses with low incidence angle XRD do not put in evidence peaks of alumina and chromina oxides. However, Raman analyses in fluorescence mode let us hardly detect the presence of alumina. This technique, very sensitive for the detection of alumina, confirmed the existence of a very thin oxide layer. The atmosphere chosen in this study and the protocol adopted for mechanical tests well prevent the specimens from surface degradation. The lower mechanical properties measured for ultrathin samples can be also attributed to surface softening, even noticed for single crystals [15]. Therefore the mechanical test rigs developed in the present study can be used not only for comparing ultrathin specimens together, but also for the determination of intrinsic mechanical properties.

The airfoil of turbine blade is mainly submitted to uniaxial load during the rotation of the turbine disk. Thermal and stress distributions are complex along the airfoil. For instance, the bottom part of the airfoil is exposed to a temperature around 900°C and a high stress. Conversely, the top part of the airfoil is exposed to a lower stress but a higher temperature (~1100°C). Whatever the temperature, stress couple, the yield stress of the interdiffusion zone is so low that this zone has to be considered as a non bearing zone. Indeed this mechanical state is due to stress relaxation associated with the plastic deformation of the I.Z. strata. This experimental fact validate the hypothesis made a few decades ago dealing with the calculation of an effective stress corrected after rupture [16].

## 6 Conclusion

In conclusion, a method of mechanical and physical local characterization at different temperatures has been developed in this work. First tests on ultrathin specimens enable to establish a new database. This technique can be easily translated to the characterization of other functionally graded materials (coated materials, surface damaged by oxidation or corrosion, surface treatment, machining, etc.). Mechanical tests have also been carried out on materials coming from real turbine blades as well. Techniques developed in this study offers new prospects in terms of local mechanical characterization. This approach appears to be necessary to implement real flow rules in the modeling process of functionally graded materials at the micrometric scale.

## Acknowledgement

The authors are particularly grateful to Turbomeca–SAFRAN group for providing the material. This work was part of a research program supported by DGA involving Snecma-SAFRAN group, Turbomeca-SAFRAN, ONERA, CEAT and CNRS laboratories (Mines Paris Tech, Institut P’-ENSMA, LMT-Cachan, LMS-X, CIRIMAT-ENSIACET).

## References

- [1] Caron, P., Le développement des compositions pour superalliages monocristallins. in Colloque National Superalliage Monocristallin (Seilh (France)), 65-76, 1995
- [2] Reed, R.C., et al., Alloys-By-Design: Application to nickel-based single crystal superalloys. *Acta Mater.*, 57, 5898–5913, 2009
- [3] Rhys-Jones, T.N., Coatings for blade and vane applications in gas turbines. *Corros. Sci.*, 29, 623-646, 1989
- [4] Yanar, N.M., et al., Degradation of EBPVD YSZ Thermal Barrier Coatings on Platinum Aluminide and NiCoCrAlY Bond Coats During High Temperature Exposure. in *Proc. of the Turbine Forum : Advanced Coatings for High Temperatures*, Forum of Technology, section 14 (Dorsten, Germany), 2002
- [5] Pan, D., et al., Evolution of a diffusion aluminide bond coat for thermal barrier coatings during thermal cycling. *Acta Mater.*, 51, 2205-2217, 2003
- [6] Hemker, K.J., et al., Characterizing the microstructure and mechanical behavior of a two-phase NiCoCrAlY bond coat for thermal barrier systems. *Mat. Sci. Eng. A-Struct.*, 483-484, 727-730, 2008
- [7] Majerus, P., Neue Verfahren zur Analyse des Verformungs- und Schädigungsverhaltens von MCrAlY Schichten im Wärmedämmschichtsystem. PhD Thesis (RWTH Aachen), 159, 2003
- [8] Passilly, B., et al., High temperature instrumented microindentation : applications to thermal barrier coating constituent materials. *Phi. Mag. A.*, 86, 5739-5752, 2003
- [9] Hüttner, R., et al., First creep results on thin-walled single-crystal superalloys. *Mat. Sci. Eng. A Struct.*, 510 - 511, 307-311, 2009
- [10] Zafir, A., et al., Effect of Strain Rate on Ductile-to-Brittle Transition Temperature of a Free-Standing Pt-Aluminide Bond Coat. *Metall. Mater. Trans. A*, 42, 1431-1434, 2011
- [11] Bensch, M., et al., Secondary Creep of Thin-Walled Specimens Affected by Oxidation. in *Superalloys 2012* (Champion, Pennsylvania), 387-94, 2012
- [12] Biermann, H., et al., Rafting in monocrystalline nickel-base superalloys induced by shot-peening. *Scripta Mater.*, 43, 807-812, 2000
- [13] Zupan, M., et al., Development of High-temperature Microsample Testing. *Exp. Mech.*, 41, 242-247, 2011
- [14] Boudot, A., Propriétés des revêtements de protection haute température pour pales de turbine haute pression. PhD Thesis (INP-Toulouse), 145, 1999
- [15] Mughrabi, H., Some consequences of surface and size effects in plastically deformed copper single crystals. *Phys. Status Solidi B*, 44, 391-402, 1971
- [16] Veys, J.M., Contribution à l'étude de l'influence des revêtements protecteurs sur les propriétés mécaniques des superalliages pour aubes CMSX2 et COTAC784. PhD Thesis (Poitiers University), 219, 1987