Digital Image Correlation method for monitoring tensile tests at high temperature. Part 1: comparing the speckle quality produced by various methods at high temperature

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Résumé

L'objectif globale de cet étude est de développer une méthodologie afin d'améliorer la mesure cinématique pendant des essais mécaniques sur le matériau d'alliage de titane dans condition d'emboutissage à chaud. Afin de contribuer à l'optimisation de la méthode corrélation d'image (DIC) dans ce contexte spécifique, la génération de mouchetis adapté aux conditions de mesure est proposée. Dans un premier temps, les mouchetis préparés par plusieurs méthodes différentes sont comparés en termes de la qualité. Pour pouvoir comparer objectivement, un mouchetis numérique est systématiquement utilisé. Dans un deuxième temps, les images de surfaces mouchetées sont capturées à différentes températures et puis comparées selon plusieurs critères variées par exemple le contraste, le niveau de gris, etc…. A température ambiante, le premier résultat montre que le mouchetis préparé par la méthode anodisation présente le meilleur contraste. Cependant, à haute température, cette observation est plus valable à cause de l'oxydation développée. Troisièmement, l'adhésion du mouchetis sur la surface des éprouvettes en alliage de titane Ti-6Al-4V en grande déformation est évaluée. Mouchetis préparé par la méthode gravure laser résiste à la rupture. En même temps, le mouchetis préparé par la peinture s'écaille au début de la déformation plastique. En conclusion, une suggestion de choix du mouchetis adapé aux conditions de mise en forme à chaud est proposée.

Abstract

The aim of the global study is to develop a technique to improve kinematic fields measurements during mechanical tests performed on Ti-6Al-4V alloy under hot forming conditions. In order to contribute to the optimization of Digital Image Correlation (DIC) procedures for this specific context, the generation of a suitable speckle is studied. Firstly, speckle made of different fabrication techniques is compared in terms of speckle quality. To proceed objectively, the same synthetic pattern is systematically used. Secondly, images of the speckled surfaces are then captured at different temperatures and compared in terms of contrast, grey level distribution, etc. At room temperature, the first results show that speckle made by direct anodization method lead to a better contrast. However, at high temperature, because the oxidation layer develops, this observation no longer holds true. Thirdly, the adhesion of speckle produced by different methods on Ti-6Al-4V surfaces is evaluated at high temperatures. Large strain tensile tests are performed. Speckle made by laser methods withstands large strains until necking. Meanwhile, speckle made by painting methods flakes off when the plasticity develops. In conclusion, a guideline for making a speckle suitable to perform mechanical tests under hot forming conditions is suggested.

Mots clefs : Digital Image Correlation, speckle, tensile test, high temperature

1 Introduction

Ti-6Al-4V titanium alloy is widely used in aerostructures because of excellent performance: high mechanical specific resistance and low corrosion at high temperature. Some aerostructures such as pylon or air inlet present complex shapes. They are usually manufactured by hot stamping in which temperatures are generally from 600°C to 800°C. One of the challenges of this process is to control the distortion of the part after the entire procedure [1].

In order to reduce manufacturing costs and limit the number of readjustments induced in this context, but more generally in hot forming processes, numerical simulations are usually used to predict final shape. To improve the thermo-mechanical modelling adopted, the identification of constitutive parameters and the validation of the numerical simulations would benefit from continuous full-field kinematic measurement during different production steps.

Since the 1980s, because of its ease of use and versatility, Digital Image Correlation (DIC) has established itself as the reference method to retrieve such information [2]. Although this method is relatively mastered at room temperature, it is more complex to implement at high temperature (typically from 600°C to 800°C). This is due to a number of factors, including the mirage effect (phenomena of gradient of refractive index), the loss of image contrast and the speckle flaking observed during thermo-mechanical experiments.

This study focusses on fabrication methods of speckle, its characteristics at high temperature and its adherence to the part when subjected to large strains. In DIC method, speckle characteristics are ones of the most important factors reducing noise and increasing the measurement resolution. Speckle should meet some criteria such as speckle size [3], distance between speckle patterns [4], speckle randomness [5], speckle density [6] to gain resolution, high contrast and low noise for reducing errors. Above these requirements, speckle must ensure good adherence on material's surface in case of performing mechanical tests. Finding speckle methods that satisfy such requirements at elevate temperature under high deformation is challenging. In practice, speckle can be made by many preparations techniques e.g. high temperature painting, laser engraving, etc. Indeed, Table 1 presents different speckle methods carried out in mechanical tests in the range of hot stamping temperature and some observations.

Year	T (°C)	Type of test	Speckle method	Speckle size	Observation	Ref.
1990	600	Tensile test	Manifold paint	0.2 - 2mm	Maximum deformation measurement at 0.2%	[7]
1996	650	Tensile test	Boron Nitride and Aluminium Oxide based ceramic	100µm	Speckle washed-out at high strain	[8]
2010	600	Tensile test	Commercial black high temperature paint	NG[1]	Measurement of expansion coefficient	[9]
2014	800	Tensile test	Rust-Oleum paint	NG	Speckle washed out at high strain	[10]
2015	450	Tensile test	Toner and laser printer	0.3-1.2 mm	Speckle wash out at 14% of deformation	[11]
2016	650 -730	Hot stamping	Black and white high temperature paint	NG	Speckle degraded because of high friction with tools	[12]
2018	400 -750	Tensile test	High temperature paint and anodization method	NG	Speckle made of painting washed out at high deformation	[13]

Table 1: Summary of speckle method used in mechanical tests in the range of hot stamping temperatures (from 400°C to 800°C)

Previous studies show that speckle made by painting methods has the speckle size more than 10 pixels with resolution is about 20-50 µm/pixel [5]. Furthermore, speckle subjected to high strain is washed out making kinematic measurement impossible. This is also observed by many authors cited in Table 1. Recently, Sirvin et al. [13,14] and Vautrot et al. [15] developed the electrochemical marking: anodization on metallic samples allowing large strains measurements. The permanent speckle made from thin oxide layer can follow deformation of material until necking. Although speckle size was large (the magnification is more than 20 pixels equivalent to speckle diameter of 1 mm), this technique suggests a new perspective for speckle method at high temperature. Hu et al. [16] used laser to mark speckle patterns on metallic surfaces. Speckle size can reach down to 5 µm thanks to high precision of laser source. However, to our knowledge, the impact of this process on mechanical behaviour has not yet been documented.

The purpose of this study is to suggest a guideline for the choice of a speckle deposition method adapted to hot stamping temperatures. First, in section “Materials and methods”, the speckle generator, the mask fabrication, the speckle fabrication methods and the criteria for the evaluation of speckle quality at different temperatures are detailed. Next, in section “Results”, the possibility of making speckle from different techniques followed by speckle quality at three temperatures (room temperature, 600°C and 700°C) in static condition is presented. From that, the adherence of speckle on

[1] NG : Not Given

Ti-6Al-4V titanium samples under mechanical loading will be presented. Finally, in section "Discussion", appropriate speckle fabrication methods that balance "good speckle" criteria and speckle adherence are suggested as a guideline while mechanical tests in hot stamping process are performed.

2 Materials and methods

2.1 Materials

Material used in this study is the Ti-6Al-4V titanium alloy. Its microstructure at room temperature is presented in Figure 1.

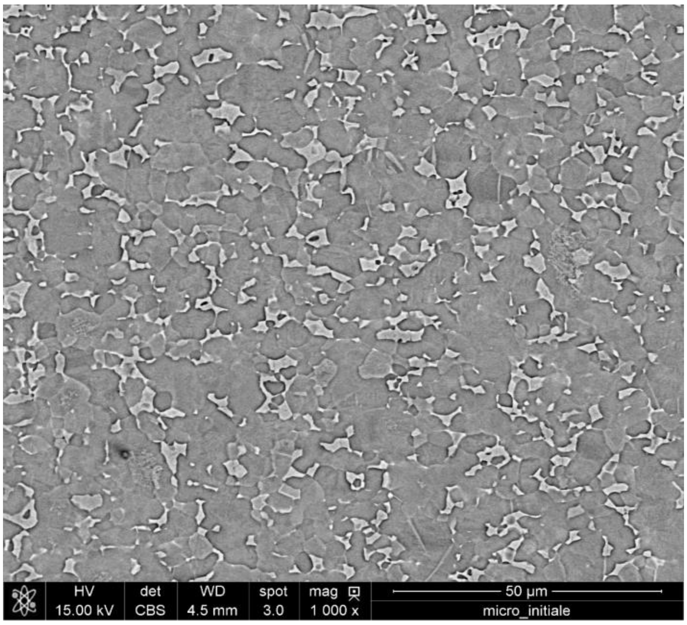

Figure 1: Microstructure of Ti-6Al-4V alloy at room temperature. Its includes the beta phase (small and bright) grains (rich in Vanadium) and the alpha phase (matrix, rich in Aluminium)

The Ti-6Al-4V titanium alloy contains two phases: the beta phase appears in small and bright colour because it is rich in Vanadium element. The alpha phase which contains Aluminium element appears in darker colour. From 400°C to 700°C, no phase transformation occurs. Its Young's modulus at high temperature is not well known. For instance, the values obtained by Chartrel [13] or Julien [17] from 500°C to 700°C vary from 90 to 60 GPa. One of the remarkable points about this material is the change of optical properties of the surface caused by the formation of Titanium dioxide. Typically, the colour of the surface changes as a function of the oxide layer thickness due to light interference phenomena. Based on this characteristic, it is possible to create speckle patterns with remarkable contrast by the anodization process.

2.2 Generation of numerical repetitive speckle

A synthetic speckle image was developed in order to objectively compare different speckle deposition techniques. The speckle generator is developed in Matlab® (Matworks, Inc., Massachusettes, USA). The speckle basically consists of a collection of randomly distributed circular dots. The main steps of the procedure adopted are: 1) a binary image that mimics the one that will be grabbed by the CCD sensor is created - the speckle pattern radius and the speckle spacing are defined 2) dots centres are then randomly distributed in the image matrix 3) speckle radius is then assigned for each dot centres such that the spacing patterns and the distance between patterns and nearest borders of matrix are respected 4) the speckle density is then calculated. If this speckle density is lower than 0.4 then blank space in matrix is filled by others patterns whose radius are equal or smaller than previous ones. The details of the algorithm are proposed in Appendix 1. Figure 2 presents a numerical speckle generated. Its density is 0.2 and the speckle pattern radius is 7 pixels equivalent to 230 µm. The smallest distance between patterns is 10 pixels, which corresponds to 320 µm.

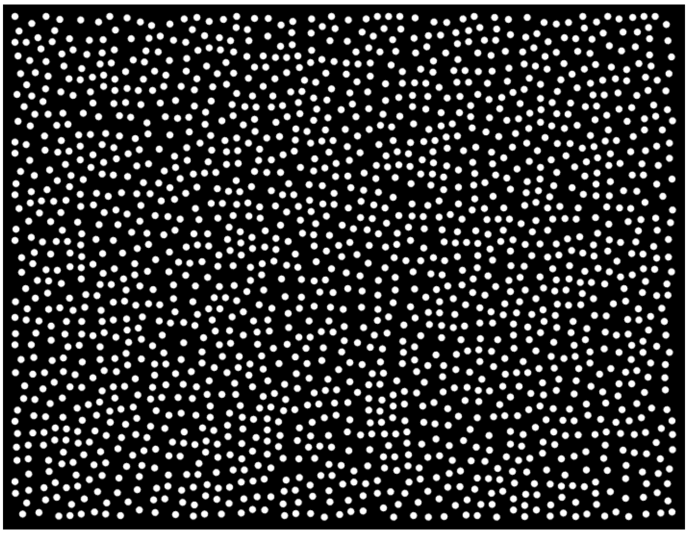

Figure 2: Numerical speckle pattern generated by the Matlab ® script. Speckle radius is 7 pixels (equivalent to 210 µm). The distance between patterns is 10 pixels (equivalent to 300 µm). The speckle density is 0.2

2.3 Speckle preparation methods

Six speckle generation methods are investigated. They consist in transferring the numerical speckle onto the Titanium surface thanks to polymer masking and laser marking. The six techniques have been chosen are:

- Painting (M1)
- Direct anodization (M2)
- Inverse anodization (M3)
- Laser engraving (M4)
- Laser and anodization combination (M5)
- Laser and painting combination (M6)

Figure 3 schematizes how these speckle techniques are processed from the synthetic one.

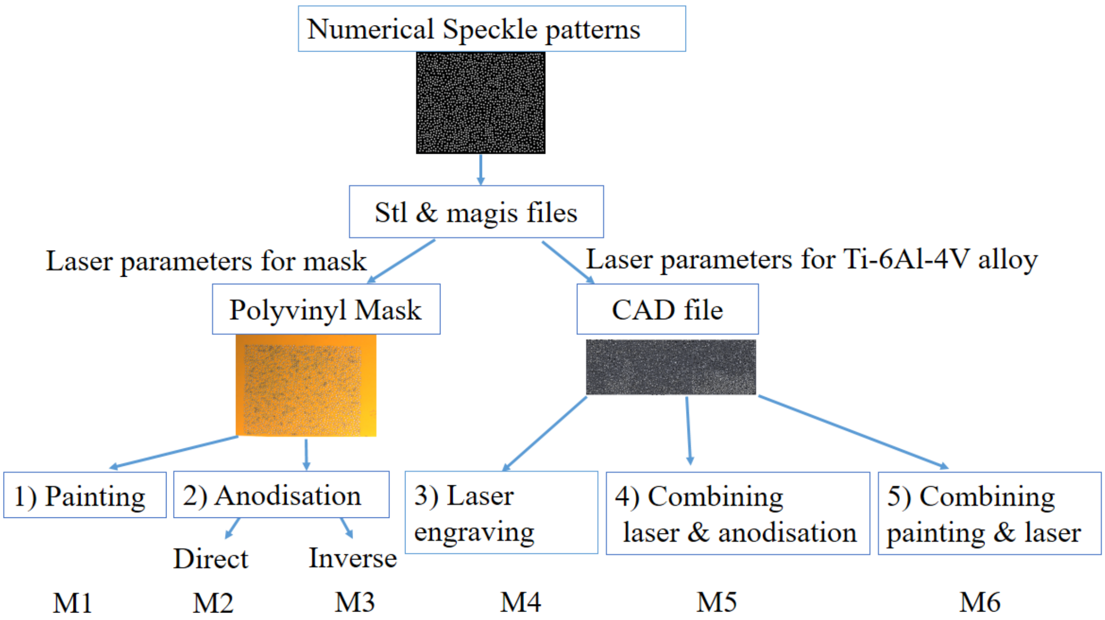

Figure 3: Principle of the six different techniques used to generate speckle patterns.

STL files created from the synthetic speckle image are then employed to manufacture the polymer mask and to mark patterns on Ti-6Al-4V samples. Those files are generated by a CAD software program, e.g. Solidworks (Dassault Systèmes, Vélizy-Villacoublay, France). A high precision laser system, namely a SLM® 125HL (SLM solution, Lübeck, Germany) is employed in order to prepare polymer mask and etch directly on Ti-6Al-4V material such that speckle patterns are similar for all six methods. On this laser machine, the laser power (P) can be adjusted from 10 W to 200 W, the laser velocity (V) from 10 to 1000 mm/s and 3 types of lasing strategy (S) are available: hatching (H), contouring (C) or a mix of hatching and contouring (H+C).

2.2.1 Mask fabrication

The aim of the polymer mask is to create similar speckle from three distinct methods: painting (M1), direct anodization (M2) and inverse anodization (M3). The material used for mask fabrication is Polyvinyl Chloride. Its commercial name is Adhesive vinyl for Cameo® whose thickness is 0.2 mm and its melting point is from 100°C to 200°C. Holes on the mask should have the same characteristics as speckle in term of speckle size, distance and density but also must not tear up during speckle fabrication steps. It means that an optimal distance between patterns, speckle size and density to gain resolutions has to be studied. To do that, first of all, an orthogonal Taguchi design of experiments is used Four factors are taken into account: i) Laser power (P), ii) laser velocity (V), iii) lasing strategy (S) and iv) speckle radius (Rc). Three levels are examined for each factor [18]. A speckle pattern is also repeated three times to test its reproducibility. The resulting Taguchi array (with 3-level-4-factors) is presented and encoded in Table 2.

Encode ID	**Level**	**Parameter**			
		Power P [W]	Velocity V [mm/s]	Lasing strategy S	Designed radius Rc [µm]
	-1	10	10	C +H	60
	0	55	80	H	106
	1	100	150	C	150

Table 2: Orthogonal Taguchi with 3 levels-4 factors in order make speckle patterns on polymer mask

After perforating the mask with the laser, an image of the mask is captured by a Leica DMS300 optical microscopy (Leica, Wetzlar, Germany). The radius of the circular holes generated is estimated using an image processing by Matlab® [19]. The response of the design of experiments is defined as the difference between the measured and the designed radius of the speckle holes:dR:

$$dR = R_{measured} - R_{designed} \quad (1)$$

2.2.2 Speckle fabrication procedures

Once the polymer mask is prepared, the next step is to apply the mask on the titanium alloy in order to produce the speckle patterns. The process is then inspired by the classic lithography technique [20]. In the anodization process, a sulphuric acid solution of 1g/l and a voltage generator are used. They allow generating different colours on Ti-6Al-4V specimens. Three speckle processes have been tested and are described in the following:

- Painting (M1): the specimen is first coated with a white paint Vitcas High temperature 800°C (Vitcas Ltd, Bristol, GB) layer to create a white background. Then the polymer mask is directly stuck on sample. A black paint Rust-Oleum High resistance temperature 750°C (Rust-Oleum, Illinois, USA) is sprayed on the stencil to create the black dots. Finally, the mask is removed carefully without smudging.
- Direct anodization (M2): the polymer mask is first stuck directly on sample. Then the Ti-6Al-4V sample is anodized in an acid solution for 30s to generate black-on white speckle patterns.
- Inverse anodization (M3): the polymer mask is first glued directly on sample. Next, the stencil is coated with a protective layer, namely Jelt (RS component, Beauvais, France). Once the mask removed, the remaining Jelt dots protect the underlying titanium surfaces from the acid solution. Samples are then anodized in acid solution for 30s to prepare white-on-black patterns. Finally the Jelt residues are removed using an acetone solvent.

Table 3 lists different kinds of paints employed in our study and their characteristics.

Purpose	**Components**	**Commercial name**	**Characteristics**
White high temperature paint	Silicone resin	Vitcas high temperature at 800°C	Hardening temperature at 160°C
Black high temperature	N-/ iso-/cyclo-alkanes C9-C11	Rust-Oleum® High temperature at 750°C	Boiling point at 160°C
Protection polish coating	Heavy disulphide naphtha	Jelt®	Hardening at room temperature

Table 3: Materials used for speckle preparation methods

For the next three methods, laser engraving (M4), laser and anodization combination (M5) and laser and paint combination (M6), the *.stl* file is directly imported in the laser machine. The speckle pattern is then engraved directly on the Ti-6Al-4V specimens. Note also that for M5 and M6 methods, prior to engraving, surfaces are covered with a white paint or a thin oxide layer by anodization.
The same laser parameters are chosen for three techniques: power P = 20 W, velocity V = 150 mm/s, lasing strategy Hatching (H) and Contouring (C). To ensure that the marking does not modify significantly the mechanical behaviour of material, the roughness, the microstructure and the rigidity of Ti-6Al-4V alloy after treatment are carefully studied. The roughness is measured using a confocal profilometer Altisurf©520 (Altimet, Marin, France). Roughness parameters are processed by MoutainsMap® (DigitalSurf, Besançon, France). The microstructure on cross section view of Ti-6Al-4V alloy after the treatment is observed by Scanning Electronic Microscopy (SEM) (FEI™, Oregon, USA) with Back-Scattered Electron (BSE) detection mode under an acceleration tension of 15keV. Tensile test is performed using MTS125 hydraulic testing machine (MTS, Minneapolis, USA) with a cell load of 50kN. The stress-strain engineering curves of titanium samples before and after treatment are compared and the adherence of speckle on material's surface is evaluated.

2.4 Evaluation of speckle quality at different temperatures

Speckle images are recorded thanks to a DIC system. The equipment used is detailed in Table 4.

Purposes and devices	**Commercial name**	**Characteristic**
Distance Camera-Object		70 cm
Camera lens	Tamron	Macro with f= 80-300mm
CCD sensor	Pike 505F/B	Resolution 2452 pixels x 2052 pixels
Lighting	Dedolight	White light with maximum power of 200W
Capturing images	VIC-Snap	Frequency of 5Hz
Measuring kinematic field	VIC-2D	2D measurement
	• Algorithm	Subset-based algorithm
	• Subset size	33 pixels
	• Step	8 pixels
	• Correlation Method	Zero Normalized Square Least Correlation (ZNSSC)
	• Filter size	15 pixels

Table 4: List of elements employed in DIC system

Speckle images are recorded in static conditions (here, no mechanical load is applied) at room temperature and at high temperatures, namely at 600°C and 700°C, after 2h30 of heating. Time amount corresponds to heating time needed to realize mechanical tests. Since no load is applied on sample, speckle patterns are prepared on Ti-6Al-4V 25 mm x 8 mm x 1.6 mm specimens. Images from the 6 speckle generation methods are then compared in terms of speckle quality criteria: a) average speckle size r [pixels], b) average speckle spacing d [pixels], c) maximum and minimum values of grey level, d) speckle density [6], e) Mean Intensity Gradient (MIG) and f) Noise [21]. These first three criteria are calculated based on a morphology algorithm [19] and a Delaunay triangulation algorithm whose built-in functions are available in the Image Processing Toolbox of Matlab®. The density of the speckle pattern is defined as ratio of the number of 1-bit pixel and total number of pixels in the ZOI. The MIG criterion is calculated by using formulas suggested by Pan et al. [21]:

$$MIG = \sum_{i=1}^{N} \sum_{j=1}^{M} \left| \nabla f(x_i, y_j) \right| \quad (2)$$

where $f(x_i, y_j)$: grey level intensity of pixel with coordinate x_i, y_j; ∇: nabla operator, M and N are dimensions (number of pixels) in the Zone of Interest (ZOI). Noise is calculated by using equation:

$$\sigma = \sqrt{\sum_{i=1}^{N} \sum_{j=1}^{M} \left(f(x_i, y_j)_{t1} - f(x_i, y_j)_{t2} \right)^2} \quad (3)$$

where $f(x_i, y_j)_{t1}$, $f(x_i, y_j)_{t1}$ are grey level of pixel with coordinate x_i, y_j at time t1 and t2, respectively. N, M are dimensions (in pixels) in the ZOI.

2.5 Evaluation of the speckle adherence at high temperatures

Tensile tests are carried out until rupture at 600°C. Standard tensile tests with a planar geometry and a strain rate of 10^{-3} s^{-1} are performed [13]. A 2D-camera records images to detect the flaking moment with a frame rate of 5 fps. Figure 4a illustrates the experimental set up and Figure 4b presents an image of the Ti-6Al-4V sample throughout a crystallized silica window.

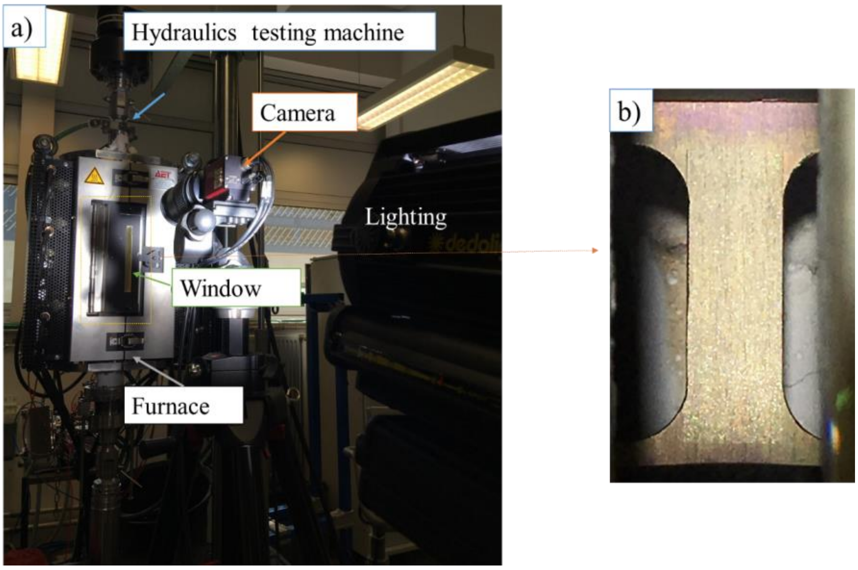

Figure 4: a) Overview of the experimental set up for evaluating the adherence of speckles on the Ti-6Al-4V alloy at 600°C, b) Image of the Ti-6Al-4V specimen speckled by combining laser and anodization technique (M5) at 600°C

3 Results

3.1 Possibility of speckle fabrication by six different methods

3.1.1 Mask preparation and speckle image prepared by painting (M1), direct anodization (M2) and inverse anodization (M3) techniques

The aim of the design of experiments procedure is to find appropriate laser parameters that allow holes to be made in the polymer mask in accordance with the synthetic speckle. Meanwhile, the polymer mask must resist to the mechanical loads induced by its manipulation during different speckle fabrication steps. As presented in the section 2.2.1, a design of experiments with four factors P, V, S, Rc is followed. The Pareto diagram (Fig. 5) presents dR values as a function of each influence factor and the cumulative percentage of each factor.

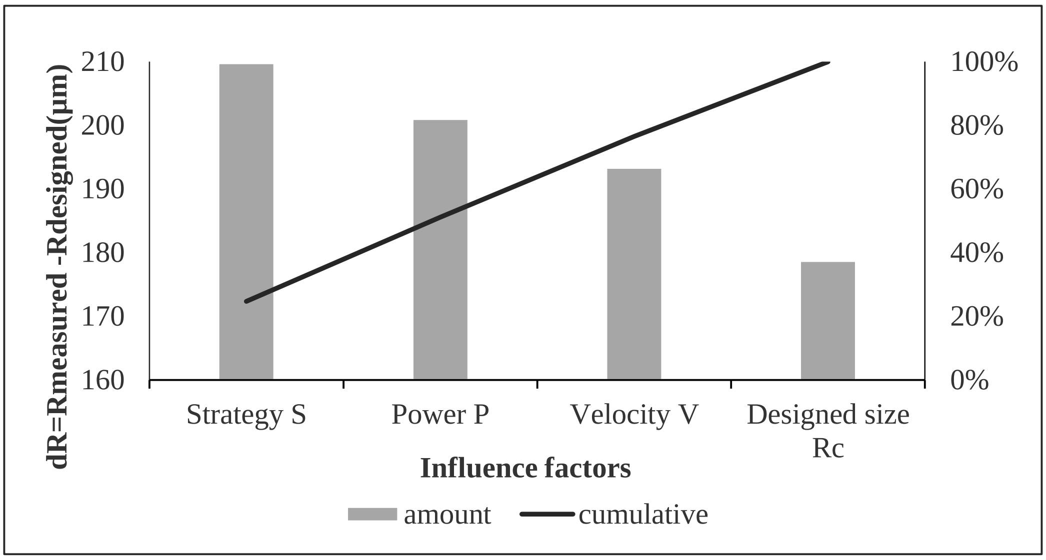

Figure 5: Pareto diagram on amount and cumulative percentage of four factors influence on response values dR It is shown that the four factors have the same order of magnitude of dR.

It is likely that the four factors have a similar influence on the size of speckle holes. In order to predict value of dR depending on factors influence, the model of linear regression available on Excel Microsoft Office® (Windows, Seatle, USA) is employed to correlate dR = f(P, V, S, Rc). The predictive model shows a coefficient of correlation $R^2 = 0.63$ meaning that the model is reasonably reproducing the experimental data. The value of dR is written as a mathematical formula:

$$dR = 111 - 45 \times S + 12 \times V + 49 \times P + 103 \times Rc \quad (4)$$

where S is the lasing strategy; V is the laser velocity (mm/s), P is the laser Power (W) and Rc is the designed radius (µm).

A comparison between dR measured and dR predicted by the linear regression is presented in Figure 6.

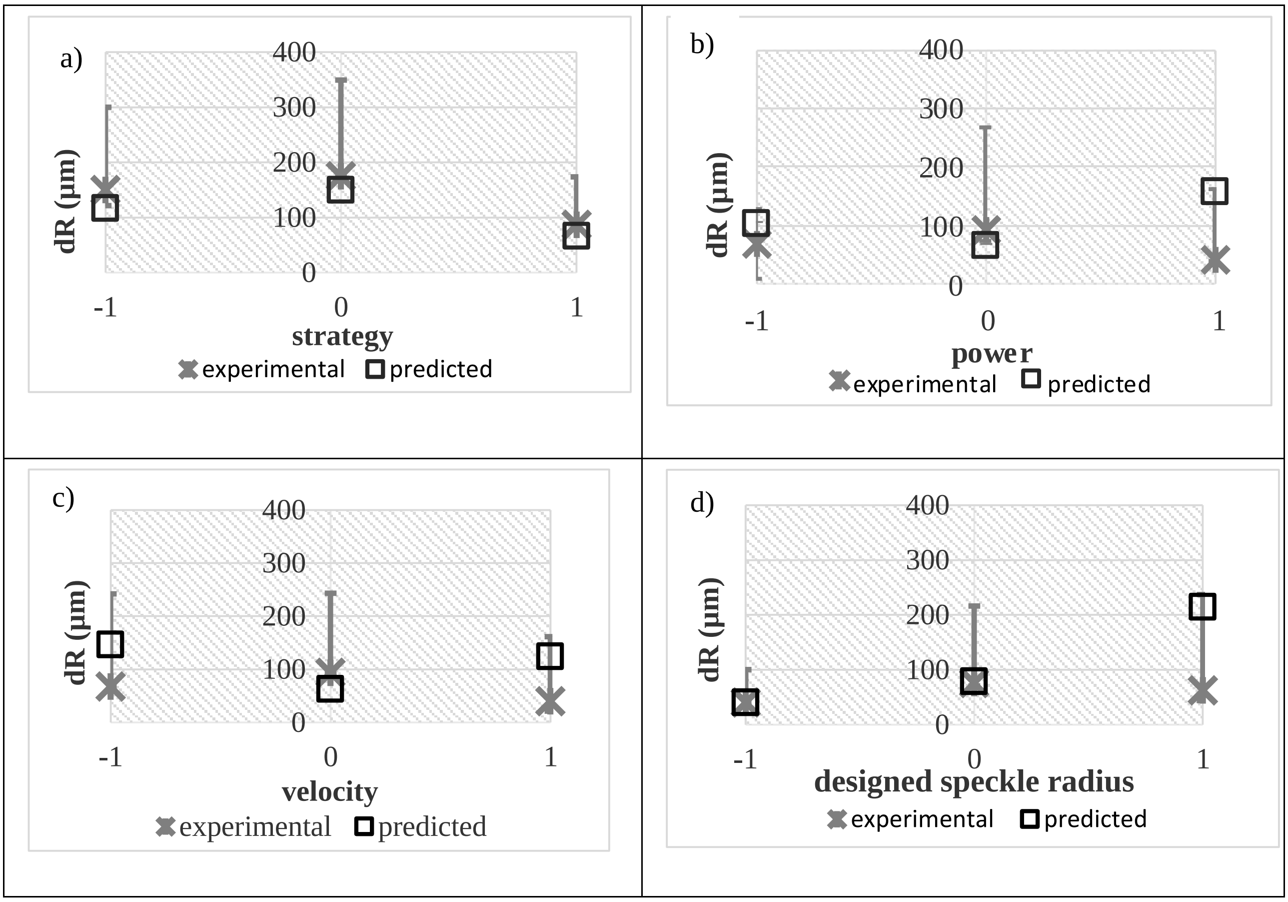

Figure 6: Influence of factors: a) strategies (S), b) power (P), c) velocity of laser (V), d) designed speckle radius (Rc) on the dR value (difference between the radius measured and the designed one). In each diagram, the experimental points (in grey star marker) are plotted with predicted point by linear regression model (in black square markers)

Predicted results match fairly well the experimental one. Some of the experimental points are however highly dispersed and far away from the predicted ones because of burnt holes. This can be explained by the important energy of laser. Figure 6a- Figure 6d present the influence of factors Strategy (S), Power (P), Velocity (V) and designe radius (Rc) on the response values dR. In our study, we look for laser parameters for which dR tends toward 0. Figure 6a indicates that the strategy of mixing Hatching and Contouring H+C (corresponding to encoded level +1) gives a minimum value of dR. The speckle size is then more similar to synthetic one than other techniques. Figure 6b presents the influence of laser power on dR values. It shows that low energy (10 W) or medium energy (80 W) would allow a correct pattern on polymer. Figures 6c presents dR as a function of velocity. It indicates that medium velocity (80 mm/s) or high velocity (150 mm/s) should be chosen. Finally, small or medium speckle radius (60 µm or 105µm) provides a higher precision (Fig. 6d). By combining all possibilities of these four factors influences, three possible sets of parameters for the laser machine are identified:

1) Configuration 1: P = 55W, V = 80mm/s, S = C
2) Configuration 2: P = 55W, V = 10mm/s, S = H+C
3) Configuration 3: P = 100W, V = 80mm/s, S = H+C

However, in order not to burn the polymer, the distance between speckle patterns must be taken into account. Experimentally, a minimum distance of 320µm with configuration: P = 55 W, V = 80 mm/s, Rc = 150 µm must be respected in order to distinguish speckle patterns.

Results show that masks with holes that conform to the synthetic image can be made by respecting the parameters listed in Table 5.

Parameters	Acronym	Value
Power	P [W]	30
Velocity	V [mm/s]	80
Strategy	S	H+C
Numerical radius	Rc [μm]	100-150 μm
Minimum speckle distance	d [μm]	300 μm

Table 5: Lasing parameters used to prepare polymer mask for speckle preparation methods

Speckle patterns made from polymer mask by painting (M1), by direct anodization (M2) and by inverse anodization (M3) are presented in Figure 7 (images captured at room temperature).

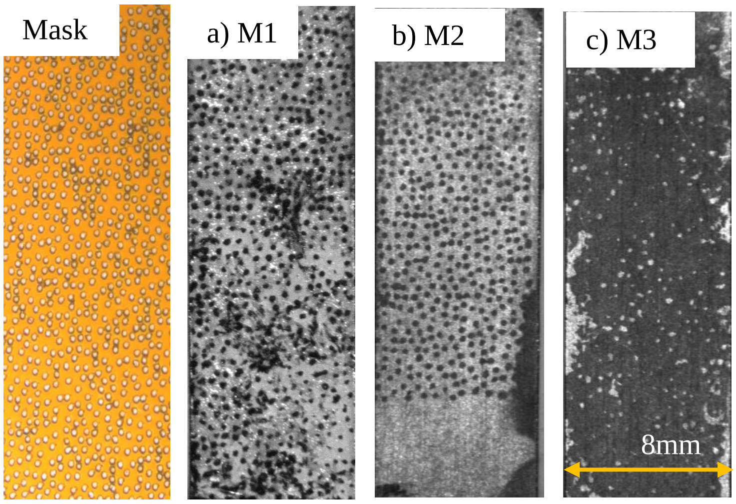

Figure 7: Polymer mask and speckle patterns prepared by techniques: a) painting (M1), b) direct anodization (M2), c) inverse anodization (M3)

3.1.2 Speckle images prepared by laser engraving method (M4), combining laser and anodization method (M5) and by combining laser and paint methods (M6)

Speckle patterns made by three laser methods, namely by: engraving method (M4), by combining laser and anodization method (M5) and by combining laser and paint methods (M6), are shown in Figure 8.

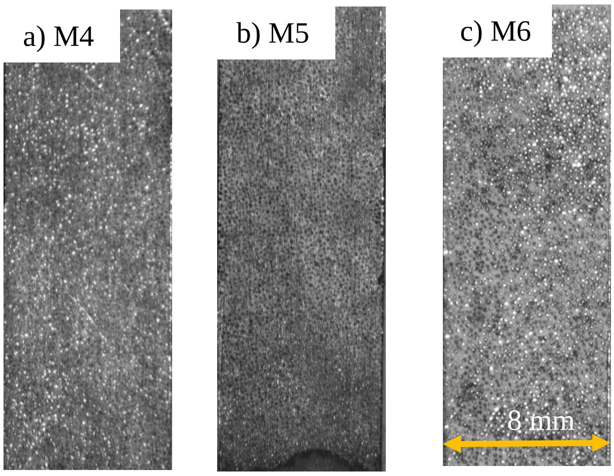

Figure 8: Speckle patterns prepared by techniques: a) laser engraving (M4), b) combining laser and anodization (M5), c) combining laser and paint (M6)

The surface roughness, the microstructure and the mechanical behaviour of the Ti-6Al-4V alloy are carefully studied as well. (Fig. 9)

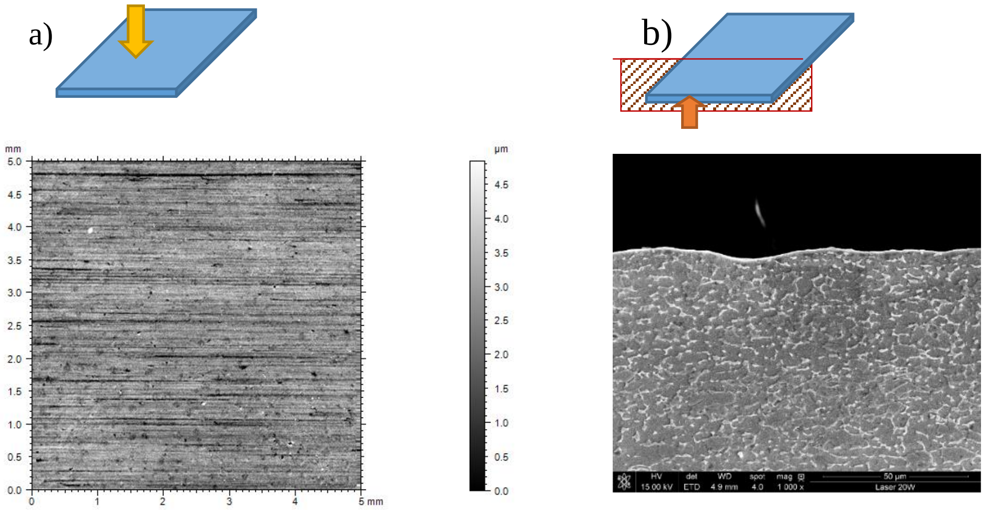

Figure 9: a) Surface roughness of Ti-6Al-4V sample, b) SEM observation on throughout thickness of Ti-6Al-4V after creating speckle patterns by laser engraving method (M4)

The roughness topography (Fig. 9a) shows a crater-like profile created on the surface of Ti-6Al-4V sample by the laser beam. Speckle patterns appears deeper (in darker colour) than average surface (in brighter colour). Ti-6Al-4V microstructure throughout thickness of sample (Fig. 9b) proves that no phase transformation occurs after the treatment. Moreover, Young's modulus of Ti-6Al-4V samples before and after treatment are also compared. Their values are 87 GPa and 95 GPa at room temperature, respectively. With the acceptable uncertainty in measurement about 10%, it is concluded that the laser engraving method does not modify significantly the microstructure and the mechanical properties of the Ti-6Al-4V alloy. The energy of 20 W and the velocity of 150 mm/s of laser system is enough for creating speckle patterns.

3.2 Quality of speckle image at different temperatures

3.2.1 Quality of the speckle image at room temperature

Speckle patterns are successfully created by six different techniques. Quantitatively, the quality of the obtained speckle images at room temperature is evaluated by speckle criteria (see Table 6).

Method	Method	Exp. time (ms)	r[pixel]	d [pixel]	Grey level	Density	MIG	Noise
M1	Painting	3.5	10±8	49± 22	[17 255]	0.6	9.7	1.6
M2	Direct anodization	2.5	4±5	30± 20	[28 255]	0.5	15.6	1.4
M3	Inverse. anodization	2.5	4±3	51± 26	[30 255]	0.1	8.3	1.4
M4	Laser	6	3±2	23± 11	[38 255]	0.2	9.9	1.2
M5	Combining laser + anodization	6	2±1	19± 10	[20 255]	0.1	10.9	1.2
M6	Combining laser + painting	4	5±5	39± 26	[34 255]	0.4	13.9	1.5

Table 6: Quantitative evaluation on speckle image at room temperature

3.2.2 Quality of the speckle image at 600°C

When six different speckles are exposed at 600°C during 2h30, it is observed that oxide layer appears on Ti-6Al-4V samples. Consequently, image quality deteriorates. Indeed, images captured (with the same exposure time than at room temperature) become darker because of oxidation (Fig.10).

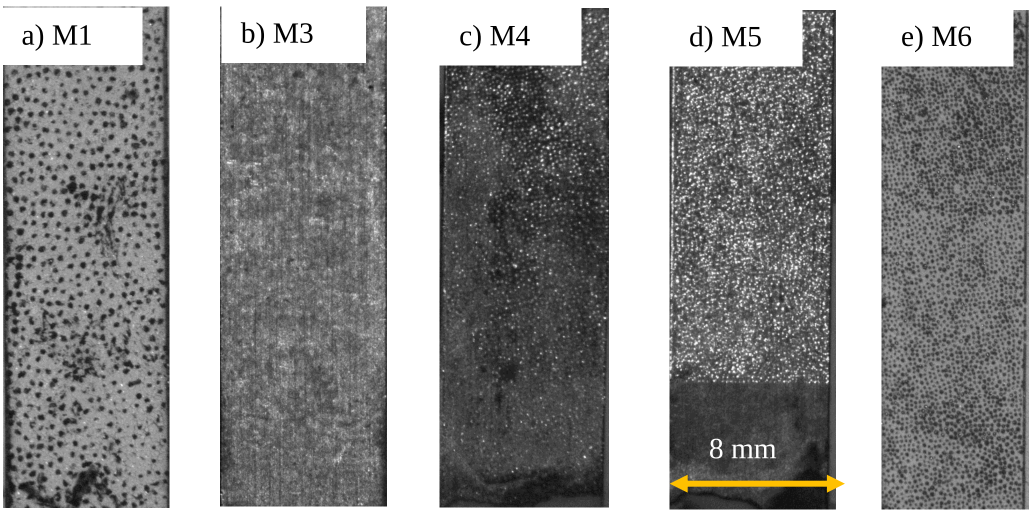

Figure 10: Speckle images captured at 600°C prepared by techniques: a) painting (M1), b) Inverse

anodization (M2), c) laser engraving (M4), d) combining laser and anodization (M5), e) combining laser and paint (M6)

Table 7 quantifies values of the speckle criteria. Indeed, with the same experimental conditions than at room temperature (lighting, exposure time, camera aperture, magnification), MIG value has reduced between 11% for M6 (combining laser and paint technique) to 67% for M2 (inverse anodization method). Interestingly, for the M5 sample (speckle made by combining laser and anodization technique), MIG value slightly increases because the reflection decreases.

Method	**Method**	**Exp. time (ms)**	**r [pixel]**	**d [pixel]**	**Grey level range**	**Density**	**MIG**	**Relative decrease of MIG**	**Noise**
M1	Painting	3.5	8±5	42±17	[22 255]	0.6	7.5	23	2.7
M2	Direct anodization	2.5	18±17	84±30	[14 255]	0.3	5.2	67	1.1.
M4	Laser	6	3±2	23± 12	[22 255]	0.1	7	29	2.1
M5	Combining laser + anodization	6	3±2	18± 9	[31 255]	0.2	16.3	0	2.1
M6	Combining laser + painting	4	3±5	19±10	[22 255]	0.3	12.4	11	1.7

Table 7: Quantitative evaluation on speckle image at 600°C

3.2.3 Quality of the speckle image at 700°C

At 700°C, except for M1 (painting) and M6 (combining laser and paint) images turn black due to a high oxidation (Fig.11).

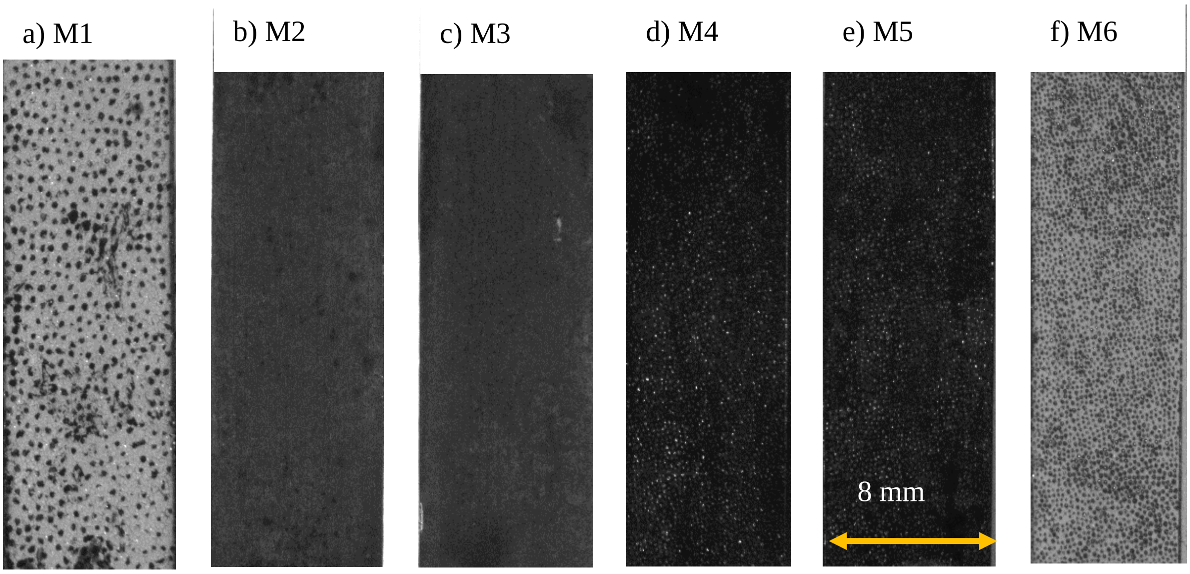

Figure 11: Speckle images captured at 700°C prepared by techniques: a) painting (M1), b) Direct anodization (M2), c) Inverse anodization (M3), d) laser engraving (M4), e) combining laser and anodization (M5), f) combining laser and paint (M6)

By looking at Table 8, it is observed that the image contrasts are reduced because of oxidation acceleration. At 700°C, the MIG criterion decreases considerably, from 23% to 76 % according to different samples. The histogram of grey level is reduced because of dark images. Noise increases because of radiation [15] and mirage effect [8,9,13,17]. Only sample M1 and M6 present a reasonable contrast.

Method	**Method**	**Exp.time (ms)**	**r [pixel]**	**d [pixel]**	**Grey level range**	**Density**	**MIG**	**Relative decrease of MIG**	**Noise**
M1	Painting	3.5	8±5	42±17	[22 255]	0.6	7.5	23	2.8
M2	Direct anodization	2.5	20±14	87±34	[28 218]	0.3	3.8	76	5.2.
M3	Inverse anodization	2.5	17±61	92±19	[28 218]	0.4	3.8	54	2.1
M4	Laser	6	2±1	25± 14	[11 255]	0.4	3.9	61	2.1
M5	Combining laser + anodization	6	2±1	20± 11	[11 255]	0.2	4.4	60	1.2
M6	Combining laser + painting	4	6±5	26±14	[19 255]	0.6	9.5	32	2.5

Table 8: Quantitative evaluation on speckle image at 700°C

MIG value can be improved by increasing the exposition time or by changing light conditions. In our study, the exposure time is increased in order to get a wider range of grey level and higher value of MIG. Table 9 shows that the grey level range, MIG values increase after increasing the exposure time for image acquisitions at 700°C. Among the six methods, the speckle made using the laser method (M4) shows the best value of MIG, thus the best contrast. The other techniques have the same order of MIG values. Noise increases because of increasing exposure time.

Method	**Method**	**Exp. time (ms)**	**Grey level range**	**MIG**	**Noise**
M1	Painting	4	[12 255]	7.5	2.8
M2	Direct anodization	18	[93 255]	10	2.1
M3	Inverse anodization	18	[95 255]	8.1	2.1
M4	Laser	7.2	[83 255]	10.9	2.8
M5	Combining laser + anodization	7.2	[85 255]	9.2	2
M6	Combining laser + paint	6	[19 255]	9.4	2.8

Table 9: Grey level range, MIG and noise values of speckle image at 700°C after increasing the exposition time

3.3 Adherence of speckle at high temperature

Four methods with the better contrast comparing with others techniques are chosen in order to evaluate the adherence of speckle on Ti-6Al-4V Titanium material at 600°C: painting (M1), laser (M4), combining laser and anodization (M5) and combining laser and paint (M6). Figure 12 presents the first wash-out of speckle on sample during tensile test at high temperature. In techniques using laser (M4) and combining anodization and laser (M5), speckle patterns are etched in materials. Therefore, they can follow the deformation of material until necking. On the contrary, speckle made by painting (M1) and by combining painting and anodization (M6) are washed out when plastic strains become large. A maximum strain of 20% was obtained before paint flakes out (ε_{max}= 0.2).

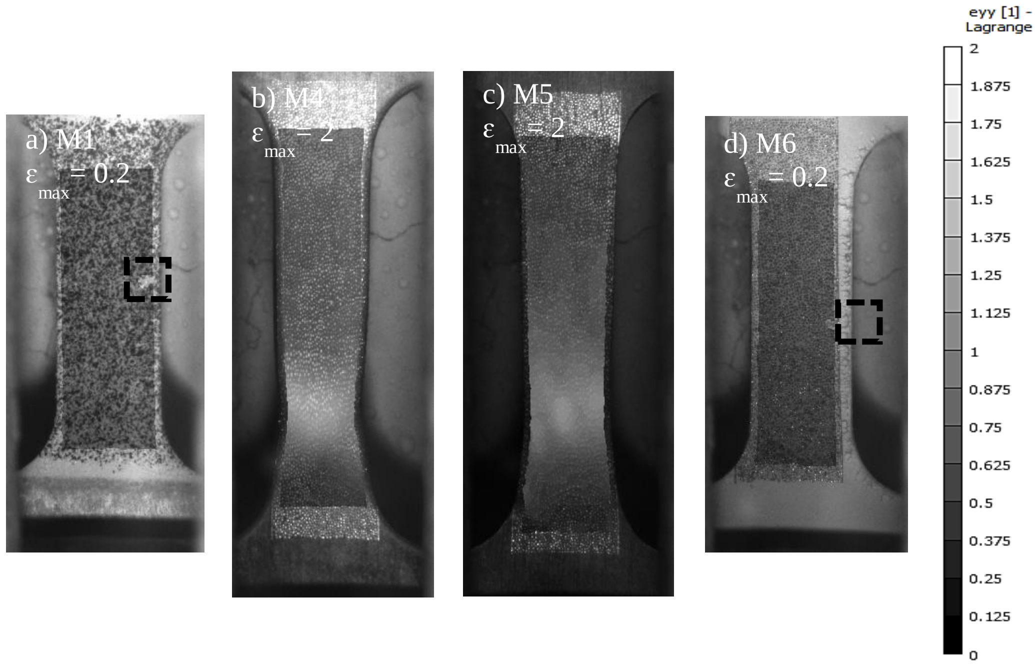

Figure 12 : Detection of speckle flaking moment on Ti-6Al-4V Titanium samples at 600°C: a) speckle made by paint (M1), b) speckle made by laser engraving (M4), c) speckle made by combining laser and anodization (M5), and d) speckle made by combining laser and paint (M6)

4 Discussion and conclusion

4.1 Possibility of making repetitive speckle

The purpose of our study is to produce repetitive speckle patterns by different speckle fabrication techniques thanks to numerical speckle model. The advantage of numerical speckle is that the speckle size, the speckle spacing, and the speckle density are controllable. At the end, this should allow a higher DIC resolution. The question is the possibility to make similar patterns by different techniques while respecting speckle criterion. It is seen in section 3.1 "Possibility of speckle fabrication by six different methods" that globally, speckle patterns are successfully made by all six techniques. They are classified by two families: The first family used polymer mask (M1, M2 and M3) while the second one used laser source (M4, M5 and M6).

Speckle patterns using polymer mask is indeed classified in "constructive" methods [5] since these three methods add materials on surface of samples: paint or oxide layer. These techniques are actually inspired from a typical UV photolithography developed by Scriven et al. [20] but at micro-scale. Actually, as far as we know, any research has been presented by using this technique applied for macro-scale patterns (its speckle radius is the order of hundred µm and applications are usually for mechanical tests). Only Mazzoleni et al. [11] used laser printer and toner transferred process for making size-controlled speckle on surface of Aluminium alloy. In his study, patterns respecting criteria in terms of size, space and density are printed on paper then transferred on the surface samples via a thermo-mechanical process. At nano-scale, equivalent techniques to our study are E-beam lithography or UV lithography technique with speckle size controlled. The average spot size of E-beam lithography method can be reach down to 4 µm that enhances displacement resolutions. However, micro-scale lithography techniques are costly and time consuming. The advantage of repetitive speckle using polymer mask is that speckle size is controllable, less costly than micro-scale techniques.

In the first family, making polymer mask is critical. It is shown in section 3.1.1 "Mask preparation and speckle image prepared by painting (M1), direct anodization (M2) and inverse anodization (M3) techniques" that by a design of experiments, the polymer mask is successfully produced and its mechanical resistance is sufficient during speckle fabrication steps. However, a minimum distance: 320 µm between speckle patterns in polymer mask fabrication must be respected. Therefore, resolution of measurement is reduced. Throughout polymer mask, patterns are printed on surface of Ti-6Al-4V alloy. Figure 7 shows that visually, patterns are successfully copied from those in polymer mask. Indeed, the shape and size of patterns made by the two methods: painting (M1) and direct anodization (M2) are more or less similar to the numerical ones (Table 6). However, due to small size of patterns and paint's viscosity, a difference between designed patterns and the real ones is observed. This is especially remarkable in specimen M3 prepared by inverse anodization method. Speckle patterns made by this technique are less visible than others. Speckle density is low (0.1) inducing a higher distance between patterns (51±26 pixels). This could be explained by a higher viscosity of coating paint comparing with black paint or water. Jelt coating is made of kerosene disulphide from processed petrol whose viscosity is relatively higher comparing with water. One technique to improve speckle density is to use airbrush with high pressure to control droplets size. Results could be improved by studying the influence of paint's viscosity on speckle size and it is suggested as a perspective of next study.

On the contrary to "constructive" techniques, speckle patterns made of laser techniques: M4, M5 and M6 can be classified in the "destructive" techniques because spots are created on surface sample. Roughness topography, SEM microstructure and the comparison between Young's modulus of Ti-6Al-4V alloy before and after lasing show that laser did not significantly change the microstructure and the mechanical behaviour of material. The advantage of laser is this allows to get similar patterns as numerical ones thanks to high precision of laser system. Indeed, numerical radius is 5 pixels and the experimental one is measured about 3 pixels. Thus the resolution on measurement would be increased. Furthermore, no additional mask will be used. Laser engraving has been recently introduced in speckle fabrication methods. Hu et al. [16] and Jones et al. [22] used laser to make speckle with size of 5µm. Our study confirmed that with an appropriate parameters of laser source, the mechanical behaviour of Ti-6Al-4V sample after lasing was not changed.

4.2 Speckle quality at various temperatures

To evaluate the speckles qualities, various criteria were used. They can be classified in two categories: a) speckle sizes including patterns radius, distance between patterns (speckle spacing) and density, b) contrast of image represented by MIG value, noise and grey level range of image.
Speckle size can improve displacement measurements. That's why an experimentalist tends to use the smallest possible speckle. According to Reu et al. [3,23], speckle size increases the resolution on displacement measurements with the condition of getting enough contrast in the matching region. Author recommends an "ideal" speckle size from 3 to 5 pixels to avoid the problem of aliasing [4] and a speckle density of 0.4-0.5 [6]. Moreover, Bornert et al. [24] studied the effect of speckle size on displacement error in the ultimate error region in subset-based DIC algorithm. Author concludes that for a given subset size, a smaller speckle radius could decrease displacement error thanks to more information contained in a subset.
Nevertheless, having small speckle is not enough. Other important criteria that allows to reduce displacement error is contrast, grey level and noise values. Pan et al. [21] demonstrates that MIG value can be considered as an effective criterion to evaluate the contrast of image. In his study, author compares mean bias error obtained from six different speckle patterns having six different MIG values. It is shown that speckle with higher MIG value produces less displacement error. The MIG criterion is also recommended by Reu et al. [23].
In summary, a "good" speckle should satisfy conditions of speckle with size of 3-5 pixels, density of 0.4-0.5, wide range of grey level and lowest distance, highest MIG and lowest noise as much as possible. Now with given values of criteria for a "good" speckle, speckle qualities made of six techniques are compared to find out the best speckle at three temperatures.
At room temperature, Table 6 shows that average patterns radius is 3-4 pixels equivalent to spot radius of 80-150 µm made by laser techniques (M4, M5 and M6). Among six techniques, speckle made of painting (M1) produces a higher spot size (10 pixels) and higher density (0.6) but low contrast. At room temperature, referring to speckle requirements, speckle made by the direct anodization technique (M2) presents the best quality since its speckle size is 4 pixels, the distance between spots is 30 pixels, the density is 0.5, MIG value is 13.9 and noise value is 1.4 (0.5% of grey level range). The highest MIG value among six speckle techniques is around 14 which is quite lower than a high contrast image (its order of magnitude is around 30 [5,21]). This could be explained by saturated pixels due to the reflection of light on surface of Ti-6Al-4V samples. Indeed, speckle images made by laser produce a crater-like shape with sharp edges (Figure 9b) that reflect coming light. One way to improve contrast is improve lighting conditions to avoid reflection effect.
At 600°C, since oxidation occurs, sample is oxidized and becomes darker. The contrast of the "best" quality of speckle made by the direct anodization technique (M2) is reduced more than 60% at 600°C. Not only M2 sample, the contrast of samples speckled by laser techniques (M4 and M6) and the painting method (M1) is reduced. Surprisingly, speckle made of combining laser and anodization (M5) increases its contrast at 600°C. The reason has not yet understood but we can explain it by the prior oxide layer before lasing. It protects samples from oxidation process at 600°C. At this temperature, speckle made by the combination of laser and anodization method (M5) presents the best quality.
At 700°C, since oxidation is accelerated, contrast of image decreases considerably. It is particularly remarkable in samples without paints (M2-M6). Paint plays a protective role that helps to reduce as much as possible the contrast of image. At 700°C, only speckle made of painting (M1) and by combining painting and laser techniques (M6) can meet "good speckle" criteria. Concerning oxidation at high temperature, according to Kumar et al. [25], it is possible to predict oxide layer thickness created on Ti-6Al-4V alloy. The idea is to correlate image contrast and oxidation to get an idea about

an image contrast value for a given oxide layer. However, with a limited time, this suggests a perspective for further study on speckle quality at high temperature.
Concerning the noise level, it is noted that noise increases with temperature, from 1.5 at room temperature to 2.8 of grey level range at 700°C. Noise increases mismatch error thus increases dispersions on measurements. This is explained by radiation and mirage effect. This is shown by other authors such as Lyons et al. [8], Pan et al. [9], Hammers et al. [10] and Vautrot et al. [15]. The noise problem caused by heat exchange between sample and surrounding environment is not the priority of this study. It will be perspectives of this study.

4.3 Assessment of adherence of speckle on Ti-6Al-4V Titanium alloy in mechanical tests at high temperature

The final and the most important criterion to assess speckle patterns is its adherence on Ti-6Al-4V surface. As showed in section 3.3 "Adherence of speckle at high temperature", we have seen that speckle made by painting methods, namely M1 and M6, are washed out since maximal deformation is around 0.2 (beginning of plastic deformation). This is also observed by Turners et al. [7] and Lyons et al. [8]. On the contrary, speckle made by laser engraving (M4) and combining laser and anodization (M5) can stand out until necking. Laser engraving method was used by Hu et al. [16] but authors studied this method in elastic region. With our study, we complete and confirm that at 600°C, laser engraving method meets criteria of "good speckle" and stand out until necking deformation.
Over 650°C, the contrast of image made by laser engraving technique is reduced. If an experimentalist would like to perform a mechanical test in elastic regime while asking for high contrast, less noise, speckle made of painting (M1) or combining laser and paint (M6) should be preferred. Furthermore, from this method, it is interesting to develop another speckle technique, for example coating method, on which Ti-6Al-4V Titanium alloy is protected and assure a good adherence of coating layer and material.

5 Conclusion

Six different speckle preparation techniques adapted to Ti-6Al-4V alloy and high temperature experiments are tested. For all techniques, the same pattern is generated thanks to a numerical speckle generator. The procedure allows comparing objectively the resulting speckle quality at different temperatures. Tensile tests are finally performed to assess the adherence of speckle made of four methods at 600°C.
The techniques can be classified into two categories. Three techniques rely on a laser cut polymer as a stencil. Three others rely directly on the laser beam to mark the surface. Results from a design of experiments showed the possibility of making a polymer mask, a critical step in making speckle patterns by constructive methods such as painting (M1), direct anodization (M2) or inverse anodization (M3). A difference between speckle size of patterns obtained by the three techniques and the synthetic one is observed. This observation is probably due to the fact that the viscosity of the paint is high and that the holes in the polymer mask are small. Speckle made of "destructive" methods, including laser engraving (M4), laser and anodization combination (M5) and combining laser and paint (M6) are nearly similar to designed one thanks to high precision of laser system.
At room temperature, speckle made by direct anodization technique (M2) leads to the "best" quality in term of size, density and contrast. At 600°C, speckle made by combining laser and anodization method (M5) ensures the best contrast and good adherence on Ti-6Al-4V surface. At 700°C, due to

accelerated oxidation of non–protected surfaces, only speckle made by painting (M1) and combining laser and paint (M6) present a higher contrast when compared with other techniques. However, speckle flakes off at large deformation. The maximum value obtained is approximately 20% ($\varepsilon_{max} \approx$ 20%).

Several interesting perspectives emerge from this study. For instance, it would be interesting : i) to investigate the properties of polymer materials used for making mask and to control the droplet size of paint in order to prepare speckle patterns closer to the mask template ii) to predict the titanium oxide layer in order to correlate the speckle image and the thickness of oxide layer at different temperature. iii) to study another speckle method that meets “good speckle” criteria and good adherence on surface, e.g. golden coating method coupled with laser.

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Appendix

A. 1: Script written in Matlab® environment for numerical speckle patterns generation

1. generation of matrix whose size is equivalent to CCD sensor
2. % begin the first iteration
   define the number of speckle patterns in the matrix for first iteration
3. define speckle size (in pixel) for first iteration
4. define speckle pattern (2 bits grey level) with size defined in step 3
5. define the distance between speckle (in pixel). This exclusion distance is an exclusion region (in square) in which next patterns generated cannot be included.
6. % make a loop that satisfy conditions:
   While i = 1: number of speckle patterns
   - 6.1 generate random number on matrix
   - 6.2 find position of maximum value on matrix
   - 6.3 define borders as grey level = 0
   - 6.4 check speckle pattern does not touch borders
   - 6.5 calculate sum of distance between position of maximum value and surrounding patterns
   - 6.6 compare the sum of distance in step 6.4 with exclusion distance

compare the sum of distance in step 6.4 with distance between speckle and nearest borders

If sum of distance < exclusion distance & If sum of distance < borders
Do add the next speckle patterns

End

7. % beginning the second iteration
   define the number of speckles for second iteration
8. find positions (coordinates) where no speckle is filled in matrix
9. define speckle size (in pixel) for second iteration
10. define speckle pattern with size defined in step 9
11. % make a loop that satisfy conditions
    While i = 1: number of positions of empty space
    11.1 compare sum of distance of empty space with exclusion distance
    compare sum of distance of empty space with borders

    If sum of distance < exclusion distance & If sum of distance < borders
    Do add the next speckle patterns

    End
12. Calculate the density by ratio between numbers of 1-bit pixel and 0-bit pixel
13. Result: 2-bit grey level speckle image.

A. 2: Calculation for speckle criteria used to evaluate quantitatively speckle image quality

Speckle size, distance between speckle patterns are calculated based on coordinates of centres and areas of patterns on image. These two parameters are deduced by passing an image processing procedure described in following steps:

1. Choose the Zone of Interest ZOI in which speckle patterns are present
2. Perform grey level histogram in the ZOI
3. Define grey level threshold in the ZOI
4. Binarize image into 2-bit grey level image
5. Remove speckle at image borders
6. If necessary, fill holes in speckle patterns with circle parker with size of 2 pixels
7. Performing an ultimate erosion to obtain markers
8. Labelling markers in step 7
9. Performing reconstruction image with markers in step 7
10. Labelling objects in step 9
11. Extracting information about coordinate centres, areas of object

    Radius of dots are calculated by assuming that object is completely round $r[pixel] = \sqrt{A/\pi}$ where A [pixel]: Area of object