Thermal sprayed protective coatings for superplastic forming ceramic dies: A monitoring system of die condition

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Superplastic forming (SPF) is an advanced sheet manufacturing process typically restricted to low volume and high value products. SPF ceramic dies have a lower production cost than conventional metal tools, and have short lead-times, but their brittle nature is a limiting factor for SPF applications. Suitable surface treatments have a remarkable effect on wear resistance; hence, the working surface of the die can be improved by the application of appropriate coatings, increasing the lifetime of the SPF ceramic dies. Thermal spray is a promising deposition method, since numerous coating solutions are achievable without expensive vacuum technology.

This paper describes a method for the investigation of surface degradation mechanisms in ceramic dies during SPF process at laboratory scale. Greater emphasis is given to the application of thermal sprayed coatings and quantification of their protection properties. At the Advanced Forming Research Centre is been designed a dedicated test rig used for simulate the die part interface at SPF condition. The aim of this study is to develop and to implement a monitoring system of working surface condition by which it is possible to predict the ceramic dies lifetime improvement for different thermal sprayed coatings.

Keywords: Superplastic forming; Ti6Al4V; Ceramic die; Protective coating; Thermal spray.

1 Introduction

Superplasticity is a deformation mode associated with the exceptional ductility that certain materials exhibit when deformed under specific strain rate and temperature [1]. Superplastic forming (SPF) process is characterised by high temperature and controlled gas pressure that deform the part sheet into the die configuration [1].

This phenomenon is employed with titanium alloys, in particular Ti6Al4V which has a wide application in aerospace structural components, chemical industry, automotive and marine [2], [3]. SPF is attracting the attention of researchers and engineers because of the benefits that the process can achieve [4]:

- Lower strength of the tooling is required;
- Complex shapes can be formed;
- Weight and material saving can be realized;
- Little or no residual stress occurs in the formed part.

Furthermore, the combination of diffusion bonding (DB) with SPF is an ingenious example of near-net shape manufacturing process, within which complex thin sandwich titanium structures can be made in a single step [4]. An analysis of the cost of the SPF/DB manufacturing process shows that the principal cost elements are the Ti6Al4V sheet and the tools that must withstand prolonged hours at 850-950°C [5].

SPF dies are commonly made of heat resistant nickel-chromium cast steels, but such materials are expensive and have very long manufacturing lead-time. The total flow time to design, cast, machine and polish a metal die is typically three to eight months [2], [6] that is a critical limiting factor for the SPF/DB process expansion to further applications. Moreover, nickel-chromium cast steels linear thermal expansion coefficients (CTEs) are larger than that of Ti6Al4V. The different CTEs will increase dimensional inaccuracy because of the larger thermal contraction of dies than that of the Ti6Al4V during cooling after the SPF process [7].

Recently, high temperature resistant steels are being replaced with emerging materials such as refractory castables that present a lower production cost and exhibit shorter lead-time [8], [9]. Those emerging materials can benefit the SPF process in terms of:

- Rapid prototyping;
- Lower die cost;
- Use of inductive heating.

Refractory castables are interesting for their cost and their mechanical properties at high temperature, but their brittle behaviour, which it is the dominant failure mechanism, is a limiting factor for the implementation in SPF manufacturing process. In order to improve ceramic dies ductility a fibre reinforced refractory castable has been studied [6].

Furthermore, due to the manufacturing mechanism principle, superplastically formed parts often suffer of large non-uniformity thickness distribution. This causes a reduction of the integral parts' property, which is cause of cracks generation, decreasing the forming limit of materials [10].

A suitable surface treatment can introduce important benefits to ceramic dies working surface, protecting the interface die-part from wearing out, and preventing the formation of cracks [8]. Moreover, considering the study carried by *Jiang et al.* [10], it is possible to design a coating for a “smart” working die surface, modulating the friction coefficient to achieve a desired thickness uniformity of the part superplastically deformed. In addition, the introduction of a coating with a desired CTE can increase the dimensional accuracy of workpieces. Also, it can make the workpiece easier to remove from the die at the end of the SPF process, avoiding die or part damages. Summarising, the development of a coating for SPF ceramic dies have a remarkable business impact and engineering research interest in terms of:

- Die lifetime improvement;
- Die manufacturing cost reduction;
- Die lead-time reduction;
- Uniformity of parts thickness distribution;

The research program aim is to understand coating capabilities in SPF applications, developing a protocol procedure to validate different coating materials and deposition methods. Thermal spray appears to be an attractive technique for the protective coating purposes.

2 Experiment set-up

During a SPF process a multitude of variables take place and govern the interactions between die and workpiece surfaces. In the SPF process (Fig.1) the sheet part is loaded and clamped between the top and bottom dies. A furnace provides the heating to achieve the superplastic temperature of the part, and argon is introduced into the die to apply the desired strain rate to deform the part. Once the deformation is complete, the part is cooled and unloaded from the die.

In a previous work carried at the AFRC [11] it was developed a test procedure to study the interaction between an heat-resistant steel die and a Ti6Al4V part. A set of conditions was developed to be broadly representative of the SPF forming cycle.

The present work it is aimed to develop a test procedure for a refractory castable die and a Ti6Al4V part. Through a dedicated test rig the experiment consists of generating a pressure between 3 to 9 MPa over a representative area at SPF temperature for titanium alloys (850-950°C).

In the recent years an outstanding effort have been made by the AFRC team in order to achieve a systematic method to analyse and monitor the die lifetime through the analysis of the scale built-up on the titanium parts after the SPF process [11]. After each run a scale arises on the titanium parts, because of different variables that have place during the SPF process: at those conditions several physical and chemical interactions happens between the die and titanium part interface.

A protocol has been created to characterize and control the metallic die lifetime during his application at SPF condition. The protocol is based on the measurement of parts surface features and their correlation with thermal images of the working interface of the die. Furthermore, a SEM/EDAX analysis is suitable for further analysis. The protocol has been validated for metallic dies. In the current work a refractory castable die has been tested to validate the protocol for ceramic materials.

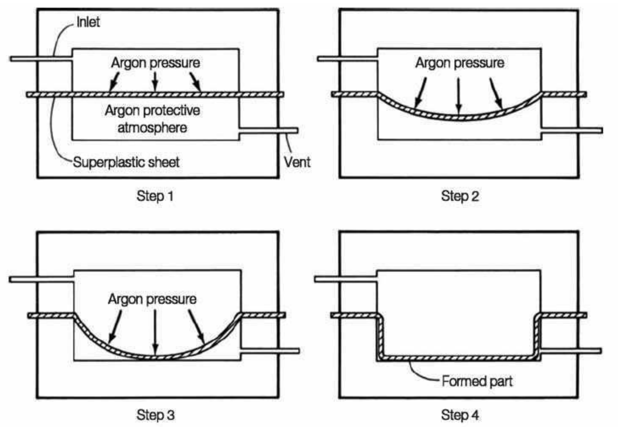

Fig.1. Schematic view of SPF process. The sheet part is loaded into the die and clamped. The inlet valve on the top die is designed for the introduction of the processing gas. The bottom die has a vent valve. Dies and part are introduced into a furnace for achieve the processing temperature. Controlling the argon pressure it is possible to induce the deformation of the part at a predetermined strain rate.

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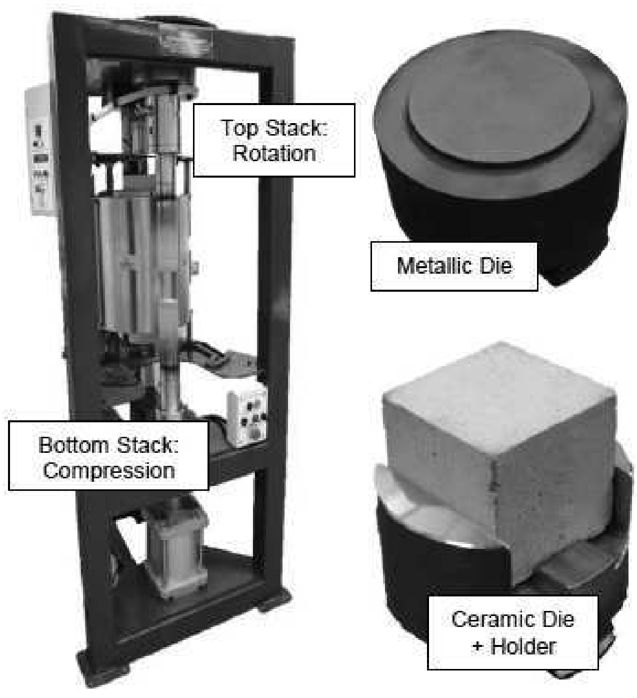

Fig.2. On the left, the test rig that simulate the SPF process. It is composed by two stacks and a cylindrical furnace. On the right: on top a tool for the test rig that simulate a metalic die during the

SPF process. On the bottom a ceramic cube and his holder that simulate a ceramic die. The test rig is now omologate to test both metalic and ceramic.

The rig has been designed to test metallic dies, but it does not exclude the ability to test others materials. For this purpose, in this work a suitable holder has been designed to fit ceramic dies into the rig. Now, the rig allows to conduct a fundamental analysis of the interface conditions of both metallic and ceramic dies.

2.1 Upgrade for test ceramic dies

The test rig (Fig.2) is a unique equipment whereby it is possible to simulate the SPF process. The rig is composed by two stacks. The bottom stack introduces the static load that simulates the gas pressure in the SPF chamber. The top stack is linked to a rotation motor that simulates the sliding of part onto the die surface during the SPF process [11].

The ceramic die material is the Ceradyne Thermo-Sil® 220. It is a fused silica castable with the addiction of silicon carbide reinforcing particles. The ceramic die for the test is simulated by small ceramic cubes of 50 mm side.

2.2 Experimental procedure test

The test is designed to replicate the die-part interface condition during the SPF of Ti6Al4V (Ti64) parts. It consists of the analysis of the working surface wear for several SPF cycles. In Fig.3 it is shown the flow chart of the experiment, which is divided into two paths: one for the Ti64 parts and one for the ceramic die. A Ti64 sheet 1mm thick is cut into sample of 80x45mm. Each Ti64 part is cleaned with water and ethanol and dried. Then a boron nitride suspension is sprayed to deposit a homogeneous anti-stick coating that help to release the part from the die after the SPF process. Using and optical microscope (cf. Section 3.1) a surface roughness inspection is carried on random selected Ti64 parts before the experiment. The average roughness is less than 1μm (micron).

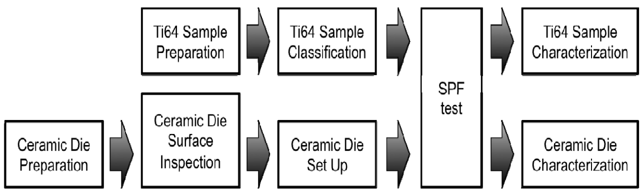

Fig.3. Experimental work flow.

The ceramic die is a Ceradyne Thermo-Sil® 220 castable cube with dimension of 50x50x50mm. Surface roughness of casted cubes is in the 5-10μm (micron) range. 2 cubes have been casted to run the experiment. Each cube simulates a ceramic die in SPF condition. The ceramic cubes have been cleaned with compressed air before to introduce them into the furnace. Furthermore, the ceramic holder, made of high temperature resistant stainless steel, has been preheated to the test temperature before the ceramic die positioning into the test rig.

The experiment has been divided into 2 runs to be able to test the repeatability of the test protocol. In the run #1 was used 22 Ti64 parts, and 10 parts were tested in run #2. The same parameters (Temperature, pressure and time) were used in each cycle (Table 1).

Table 1. Experiment parameters.

Run	**Temperature** [°C]	**Pressure** [Mpa]	**Cycle time**	**Number of cycles**
#1	850-900	2	1h	22
#2	850-900	2	1h	10

The aim of the test is to know the response of the ceramic die surface. Also, through the test it is wanted to estimate the capability of the rig simulating an SPF process with ceramic tools.

The purpose of the research project is to apply, mainly via thermal spray, protective coatings to both metallic and ceramic dies, in order to achieve a greater die lifetime minimizing surface wear.

3 Monitoring systems

3.1 Optical surface metrology

In order to achieve a monitoring protocol, surface characterisation methods are employed. It is not possible to evaluate the die/part interface interaction in situ. An optical surface metrology system has been set-up to analyse the surface morphology of parts and dies. Scans are performed with the Alicona Infinite Focus G4, an optical 3D system capable to scan and digitize the surface morphology through a differential optical z-scan. The surface maps are then analysed to identify surface features. The sequential scan of each Ti6Al4V part lets evaluate how the die surface wear affects the part surface.

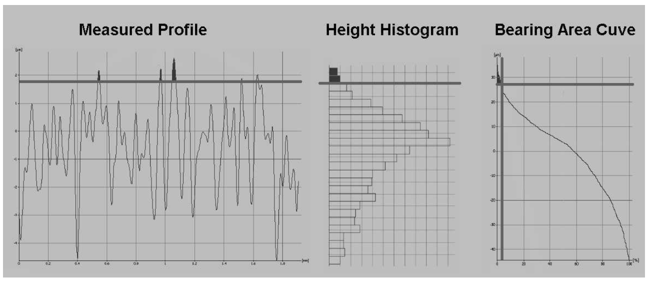

Fig.4. On the left: the profile path a selected area of a Ti64 part after the test. In the middle: the histogram of the height distribution of the profile. On the right: the Abbott-Firestone that highlights the difference of peak and valley distribution.

The surface analysis is composed by standardized profile and area measurements. Area measurements gather data from a surface, instead of a single profile path, averaging a bigger amount of values. In both the cases, the data are analysed through the Abbott-Firestone curve, known from statisticians as the accumulative distribution curve (ADC). The analysis then go further to the standard roughness parameters as Ra (average roughness), Rq (root-mean-square roughness) and Rt (peak-to-valley height) giving more details about the volume of peaks and valleys (Fig. 4).

The ceramic die surface is analysed before and after the experiment, and compared with the Ti6Al4V surface scan from each production run to determinate the rate of die surface breakdown, as already implemented for the metallic dies [11].

3.2 Chemical analysis

During the SPF process chemical reactions are expected at the die-part interface. The high temperature environment makes favourable the interaction between the titanium oxide on the workpiece surface and the parting agents on the die surface. The aim of the protective coating is to prevent or minimizing those interaction that cause workpiece defects.

The AFRC hold a Scanning Electron Microscope (SEM) equipped with the energy dispersive x-ray spectroscopy (XEDS) module for the identification of the surface composition. Chemical interaction between the constituent elements over a production cycle time at high temperature can be analysed.

Furthermore, a surface characterization of the thermal sprayed die can be achieved through the SEM/XEDS. It is possible to measure the thickness of the coating and is interaction with the substrate. The chemical analysis becomes crucial to verify the suitability of different coatings in SPF conditions. The coating should be able to be inert to the titanium alloy at high temperature, as the chemical interaction is cause of build-up scale onto the die and/or the part. Moreover, XRD analysis will be also implemented to control the solid chemistry evolution of the system die/coating/part.

3.3 Thermal imaging

The AFRC research projects fall into the Technology Readiness Levels (TRL) 3 to 6, which means that they are strictly related to industrial solutions. For this reason, it is reasonable to understand how production environment can influence the die-part interface interactions. Thermal imaging techniques have been adapted to monitor the die surface conditions during the manufacturing production cycle. A Land Instruments FTI-E 1000, 600-1000°C thermal imaging camera was installed within an SPF production facility to monitor the condition of the metallic die surface [11]. In the presented work, the same technique has been adapted to ceramic dies.

Thermal images (Fig.3) can reveal defects on the die surface that arise during several SPF cycle, due to the oxidation at the die surface. The observed temperature map is a product of different emissivity coefficients of the compounds on the surface. Thermography is implemented to monitor the oxide build-up on the die surface. Thermal images are used as a qualitative method and it is than correlated with the quantitative analysis achieved through the optical surface metrology. Such a method can be used as a quasi-in-situ monitoring method after the correlation is obtained.

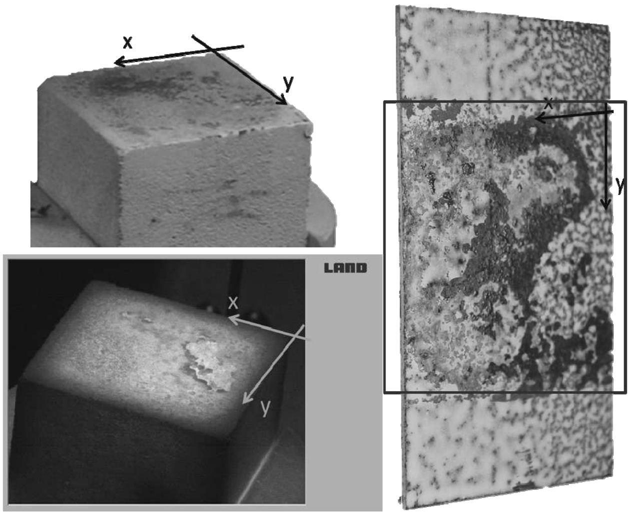

Fig.5. On the left: On the top a ceramic die after work, on the bottom a 2 colours thermal maps of the ceramic die ufter unloading of the part. Different temperature threshold values highlights areas of interest or scale build up. On the right a Ti6Al4V part after a SPF test. Some features of the part surface are visible also on the die surface through the thermal images.

4 Summary

The AFRC has built a test rig for simulate the die-part interface in SPF conditions on a laboratory scale. The rig has been designed to test metallic dies, and it is now capable to test ceramic materials too. The study of factors that affect the SPF tools lifetime is in continuous development.

A monitoring protocol for metallic and ceramic materials has been developed at the AFRC, which it is now capable to analyse die surface wearing during the SPF process. Nowadays, a release agent is used onto SPF dies, which are not provided of protective coatings for the improvement of their lifetime.

The implementation of protective coatings on SPF tools could provide a substantial economic benefit to the SPF process, making it accessible to further manufacturing applications.

Thermal spray coatings are well known and used for similar applications, and at the AFRC are available the capabilities to test those coatings in a controlled environment that simulate the SPF conditions.

The research project is intended to study the best deposition method and develop a suitable protective coating for high temperature applications, increasing the die lifetime.

From the outcomes of the research project it will be possible to implement a suitable thermal sprayed coating to SPF dies. In addition, these results could be extended to further forming processes, which are subject of interest within the AFRC and its industrial members.

Acknowledgement

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References

[1] Semiatin, S.L.: ASM Handbook vol.14B Metalworking: Sheet Forming. ASM International, (2006).

[2] Sanders, D.G.: Superplastic forming manufacturing technology moves towards the twenty-first century. Mater. Sci. Forum 304–306 (1999), pp. 805/812.

[3] Jiang, S.S. and Zhang, K.F.: Superplastic forming of Ti6Al4V alloy using ZrO2-TiO2 ceramic die with adjustable linear thermal expansion coefficient. Trans. Nonferrous Met. Soc. China 19 (2009), pp. 418/422.

[4] Leyens, C. and Peters, M.: Fabrication of Titanium Alloys. Titanium and Titanium Alloys. Wiley-VCH (2003), p. 255.

[5] Lütjering, G. and Wiliams, J.C.: Titanium. Springer Berlin Heidelberg (2007), p. 99.

[6] G. Bernhart, G. and Nazaret, F. and Cutard, T.: Fiber reinforced refractory castables for SPF toolings. Materwiss. Werksttech. 39 (2008), Issue 4–5, pp. 317/321.

[7] Jiang, S.S. and Zhang, K.F.: Study on controlling thermal expansion coefficient of ZrO2–TiO2 ceramic die for superplastic blow-forming high accuracy Ti–6Al–4V component. Mater. Des. 30 (2009), Issue 9, pp. 3904/3907.

[8] Sanders, D.G.: A Production System Using Ceramic Die Technology for Superplastic Forming. Mater. Sci. Forum 447–448 (2004), pp. 153/158.

[9] Bernhart, G. and Nazaret, F. and Cutard, T.: Fibre Reinforced Refractory Castables : an Alternative Solution for SPF Die Manufacturing. Mater. Sci. Forum 551–552 (2007), p. 37.

[10] Jiang, S.S. and Lu, Z. and He, X. and Wang, G. and Zhang, K.: Superplastic forming Ti–6Al–4V titanium alloy cylinder with near uniform thickness distribution. Trans. Nonferrous Met. Soc. China 22 (2012), pp. 472/478.

[11] Zante, R. and Knowles, C.: Advances in Monitoring Die Condition during Superplastic Forming. Key Eng. Mater. 549 (2013), pp. 557/564.