DEVELOPMENT OF A SYSTEM TO CREEP TESTS IN CONTROLLED ATMOSPHERE[1]

Danieli A. P. Reis[2]
Carlos Moura Neto[2]
Maria do Carmo A. Nono[3]
Miguel J. R. Barboza[4]
Cosme R. Moreira da Silva[5]
Francisco Piorino Neto[6]

Abstract
Because the harmful effect of the oxidation in structural materials at high temperatures was development a system to creep tests in controlled atmosphere using a conventional system to creep tests under constant load creep. The developed system was presented airtight and efficient, making possible the continuous gas flow during the test, not affecting the temperature and the load application system. Using the developed system it was studied the influence of the oxidation on creep of Ti-6Al-4V structural alloy under controlled atmosphere of argon, nitrogen and air at 500°C and 520 MPa. The creep tests presented coherent and reproductive and the system described can be used to realize creep tests in controlled atmospheres for diverse materials. The preliminary results had demonstrated that, for the alloy in question, the nitrogen atmosphere was most efficient in the increase of creep life.
Keywords: Creep; Oxidation; Ti-6Al-4V.

DESENVOLVIMENTO DE UM SISTEMA PARA ENSAIO DE FLUÊNCIA EM ATMOSFERA CONTROLADA

Resumo
Materiais com comportamento adequado em temperaturas elevadas e ambientes agressivos tornaram-se uma necessidade científica, tecnológica e economicamente viável nos dias de hoje. Estudos têm sido realizados independente de objetivos comerciais para o aprimoramento na obtenção de novas ligas e, principalmente, para a reavaliação de ligas comerciais já existentes, por meio da aquisição de dados em condições de maior severidade. Entretanto, o desenvolvimento de um sistema para ensaio de fluência em atmosfera controlada a partir de sistemas convencionais é um projeto inovador e representa um avanço no cenário tecnológico nacional e internacional, visto a necessidade de estudo da oxidação em materiais estruturais em temperaturas elevadas e ambientes agressivos, para que se possa avaliar o efeito do meio na resistência mecânica do material. Tendo em vista esta necessidade e no propósito de superá-la foi desenvolvido um sistema para ensaio de fluência em atmosfera controlada a partir de um sistema convencional de forno de fluência de tração por carga constante. O sistema desenvolvido apresentou-se hermético e eficiente, possibilitando o contínuo fluxo de gás durante o ensaio, não havendo comprometimento com a temperatura e com a movimentação do sistema. Com o sistema desenvolvido estudou-se a influência da oxidação na fluência da liga estrutural Ti-6Al-4V sob atmosfera controlada de argônio e nitrogênio a 500°C e 520 MPa. Os resultados de fluência obtidos apresentaram-se coerentes e reprodutivos podendo-se desta forma, utilizar o sistema descrito para realização de ensaios de fluência em atmosferas controladas. A atmosfera de nitrogênio foi mais eficiente na proteção da oxidação.
Palavras-chave: Fluência; Oxidação; Ti-6Al-4V.

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[2] *Instituto Tecnológico de Aeronáutica, ITA, São José dos Campos – SP – Brazil.*
[3] *Universidade de Brasília – UNB-, Brasília – DF – Brazil.*
[4] *Instituto Nacional de Pesquisas Espaciais, INPE - LAS, José dos Campos – SP – Brazil.*
[5] *Escola de Engenharia Química de Lorena, EEL-USP, Lorena – SP – Brazil.*
[6] *Departamento de Ciência e Tecnologia Aeroespacial - Instituto de Aeronáutica e Espaço, DCTA-IAE, São José dos Campos – SP – Brazil.*

1 INTRODUCTION

The phenomenon of the creep consists of the slow and gradual accumulation of deformation throughout the time, disclosing itself in all crystalline solids in favorable conditions of temperature and stress.[1] The importance technique of the phenomenon of the fracture by creep became evident from the half of the century, being recognized as one of the biggest problems of the industrial area, which had to the increasing level of requirement of the used readiness in plant of energy generation, chemical installations and in structural components developed next to the aerospace industries.[1-5]

In this way, the studies of materials used in the confection of these components demand, each technological time more improvements in the experimental assays (destructive and not destructive) and a constant search of a vast database. Such bases, associates the mathematical and computational methods, can lead to one better agreement of all the structural phenomena that can occur in the materials, when for example, submitted the stress in regimes of high temperatures.[6-8] The environment in which the test specimen is tested can exert an important influence in the resistance in high temperatures.[9-13]

The behavior of metals and alloys during the deformation in high temperatures is complex and change with parameters of processing, microstructure, applied initial stress, strain rate and temperature. Moreover, the behavior of the materials in high temperature is related with the presence of crystalline imperfections due to bigger atomic mobility and consequently to the processes that involve the phenomenon of the diffusion.[14-17]

High temperatures increase the capacity of movement of the dislocations and can activate other responsible mechanisms for the deformation process, as the possibility of operation of new systems of the slip and the deformation located throughout the grain boundary. The ambient conditions, the example of the aggressive ways exert one strong influence in the great majority of the alloys, mainly when conjugated with processes that involve the metallurgic instability, leading a structural component to a possible imperfection of premature form.[18-21]

The nature of the oxidation can have an important influence on the properties in high temperatures. A fine oxide layer normally will lead to an increase in the mechanic resistance but the intergranular penetration of oxide generally implies in a decrease of the time of rupture by creep and generally it leads the breaking of the intergranular type.[9-13] In such a way, the life in service of a material sufficiently is reduced when this must operate in atmosphere of combustion of hot gases or in corrosive ways.

Materials with behavior adequate in high temperatures and aggressive environment had become a scientific and technological necessity nowadays. Studies have been carried through independent of commercial objectives for the improvement in the attainment of new alloys and mainly for the reevaluation of existing commercial alloys already, by means of the acquisition of data in conditions of more severity.[21-24]

However, the development of a system for hot tests of the titanium alloys without covering in controlled atmosphere is an innovator project and represents an advance in the national and international technological scene, allowing to evaluate the effect of the environment in the mechanic resistance of the material.

In view of this necessity and in the intention to surpass it was development a process to hot tests of the titanium alloys in controlled atmosphere from a conventional system used to trative creep tests under constant load was developed. With the developed system it was possible of indirect form to evaluate the influence of the oxidation in the creep of the structural Ti-6Al-4V alloy under controlled atmosphere of argon and nitrogen at 500°C and 520 MPa.

2 EXPERIMENTAL PROCEDURE

A cylindrical creep furnace G28 3181-16 with three zones of heating from the Tests Laboratory of Aerospace Technology General-Command (AMR/IAE/CTA), acquired from EMEC-The Electronic and Mechanical Engineering Co. Ltd, used to creep tests in air, was adapted to realize creep tests in controlled atmospheres.

The system to the creep tests in controlled atmosphere consists of an used steel-inox tube to coat the internal part of the furnace limiting a region to the gas flow; graphite lids and seal bellows had been placed in the upper/lower part of the steel-inox tube to isolate and to prevent the loss of heat of the furnace. This seal bellows is composed of a flexible material to guarantee the movement of the system of claws during the test and to support until a maximum temperature of 220ºC (measured in this region during the test). Steel-inox flanges had been adapted in the upper/lower bellows and in the places where a tube would pass to the entrance (inferior part) and gas exit (superior part). The seal bellows was confectioned from a type of basic silica elastomer, being able to support temperature of continuous work in the range of – 55 to 230ºC.

Evaluations thermal, electric and mechanics of the used equipment had been achieved, beyond the adaptation of a new electrical system and a controller developed by the BSW Technology Industrial and Commercial Ltda with the objective to keep the work temperature in the range of 500 ± 2ºC, according to ASTM 25 E139/83 standard.[25]

Antares Software was developed in set with the BSW Technology Industrial and Commercial Ltda in previous works,[6-13] aiming the collection of relative data of the elongation of the metallic samples and measures of temperature in pre-determined periods of time.

The material chosen for the present study was hot-forged 12.7 mm diameter rod of commercial Ti-6Al-4V alloy. The microstructure (Figure 1) consists of equiaxed α grains with average size about 10 μm. The β phase is present in the α grain boundaries. Tensile testing was performed at 500°C in air. The tensile properties are summarized in Table 1 namely, 0.2% yield stress (*YS*), ultimate tensile stress (*UTS*), elongation (*EL*) and reduction of area (*RA*).

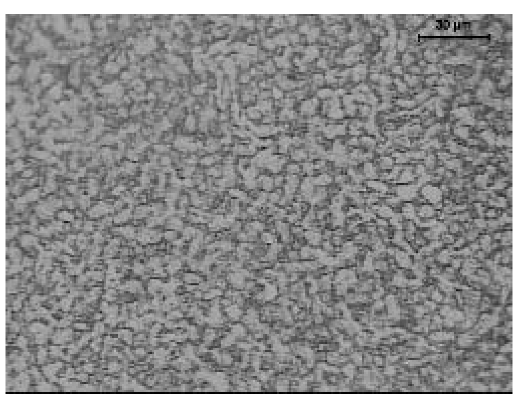

Figure 1. Micrograph of the Ti-6Al-4V alloy as-received (Kroll reagent).

Table 1. Tensile properties of Ti-6Al-4V alloy

Temperature (ºC)	YS (MPa)	UTS (MPa)	EL (%)	RA (%)
500	521	638	30	73.6

The initial creep stress levels were determined from the elevated temperature tensile properties given in Table 1. Constant load creep tests were conducted on a standard creep machine in air, nitrogen and argon atmospheres at stress level of 520 MPa at 500ºC. Samples with a gauge length of 18.5 mm and a diameter of 3.0 mm were used for all tests. The creep tests were performed according to ASTM E139 standard.[25]

3 RESULTS AND DISCUSSIONS

The Figure 2 shows a conventional system to creep test. The Figure 3 shows a schematic representation of the development system to realize hot tests of the titanium alloys in controlled atmosphere.

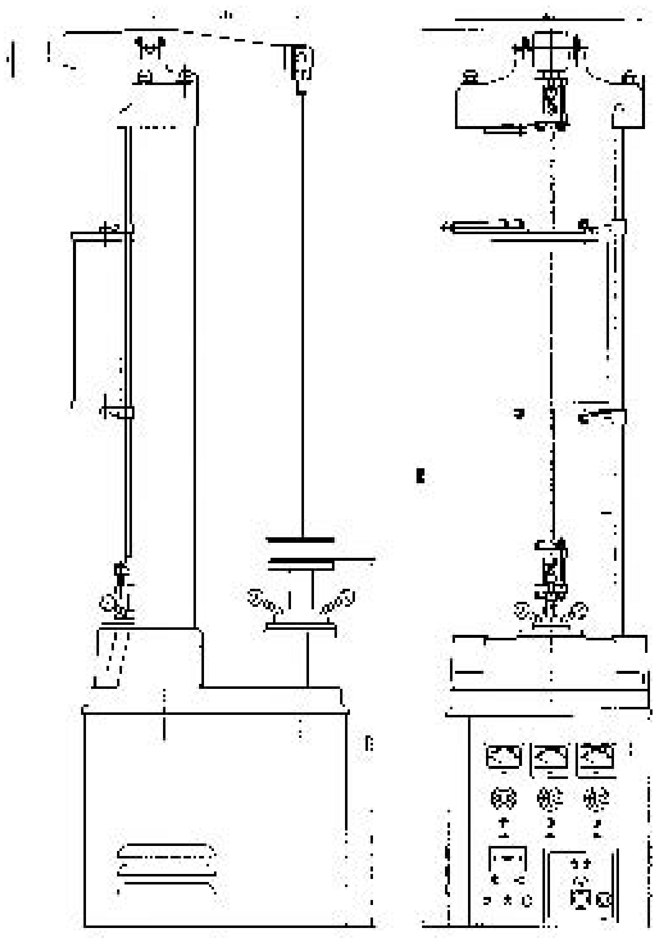

Figure 2. Conventional system to creep test.

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(a) (b)

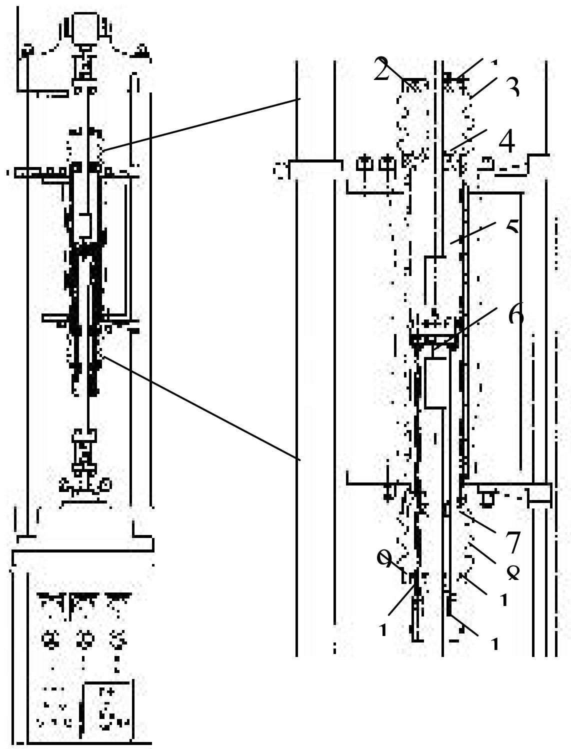

Figure 3. a) Schematic representation of the development system to realize hot tests of the titanium alloys in controlled atmosphere. b) Magnification of the sealed system of the creep furnace: 1-Superior Bellows Flange; 2-Superior O-Rings Lid; 3-Superior Bellows; 4-Superior Heat Insulator; 5-Furnace Chamber Tube; 6-Test Specimen; 7-Inferior Heat Insulator; 8-Inferior Bellows; 9-Inferior O-Rings Lid; 10-Inferior Bellows Flange; 11-Extensometer Rod; 12-LVDT Fixer.

The developed system was presented airtight and efficient, making possible the continuous gas flow during the test, not affecting the temperature and the load application system.

Representative creep curves of Ti-6Al-4V are displayed in Figure 4 in air and nitrogen and argon atmospheres. Ti-6Al-4V alloy exhibits a normal creep curve consisting well-defined primary and secondary stages. There is a relatively short initial period of decreasing primary creep rate that is associated with hardening due to the accumulation of dislocations. However, most of the creep life is dominated by a constant creep rate that is thought to be associated with a stable dislocation configuration due to recovery and hardening process.

Results from the creep tests at 500°C are summarized in Table 2, which show the values of primary creep time (t_p) defined as time to the onset of secondary creep and secondary creep rate ($\dot{\varepsilon}_s$).

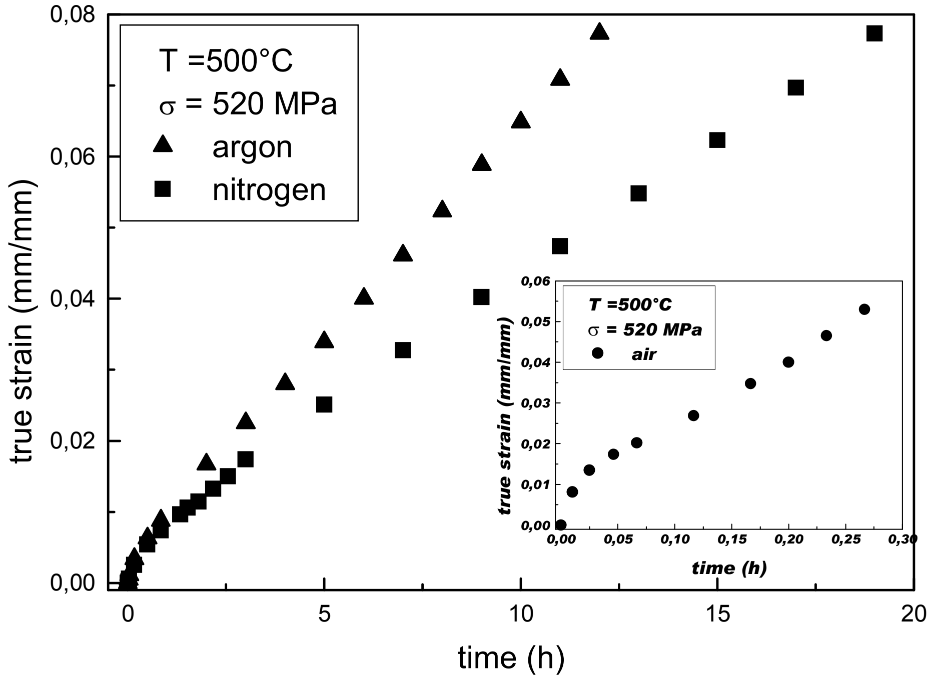

Figure 4. Typical creep curves of Ti-6Al-4V at 500ºC / 520 MPa.

Table 2. Creep data at 500°C

Atmosphere	σ (MPa)	t_p (h)	$\dot{\varepsilon}_s$ (1/h)
ar	520	0.0462	0.1596
nitrogen	520	1.8010	0.00377
argon	520	0.8333	0.00612

The results presented in Table 2 suggest that strain hardening during primary creep is dependent on the test temperature. The highest values of t_p and the reduction of the steady-state creep rate demonstrate that the higher creep resistance of Ti-6Al-4V is observed in nitrogen atmosphere. This fact is related to the hard and thin nitride surface layer formed during creep tests and the oxidation protection on creep that the nitrogen atmosphere offers to the Ti-6Al-4V alloy.

The creep tests presented coherent and reproductive and the system described can be used to realize creep tests in controlled atmospheres for diverse materials.

4 CONCLUSIONS

The creep properties of Ti-6Al-4V alloy were determined in air, nitrogen and argon atmosphere at 500°C and 520 MPa. The creep tests presented coherent and reproductive and the system described can be used to realize creep tests in controlled atmospheres for diverse materials. The higher creep resistance of Ti-6Al-4V was observed in nitrogen atmosphere.

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