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Abstract

The hot extrusion experiments have been performed in an industrial plant to point out the critical aspects of this plastic deformation technology and the related metallurgical aspects. Two types of special cross sections have been extruded, then the presence of cracks, surface defects and the homogeneity of the microstructures have been evaluated and related with the most significant aspects of the adopted technological route. The temperature of the billet and of the deformed material have been measured by an optical digital pyrometer at the different steps of the processes. A first thermal simulation based on the Fourier equation has been implemented to understand the possible influence of the heat transmission and of the thermal homogeneity on the quality of the final product. Significant indications have been reached about the relations among the adopted technological parameters and the quality of the final results. Moreover, some indications about the enthalpy developed by the industrial process of hot deformation have been determined and can be used in a next future to perform more precise simulation of the deformation process.

Riassunto

Sono state realizzate prove di estrusione a caldo in un impianto industriale al fine di mettere in evidenza gli aspetti critici di questa tecnologia di deformazione plastica e i relativi aspetti metallurgici. Sono stati estrusi due tipi di sezioni speciali e successivamente sono state valutate la presenza di cricche e di difetti superficiali e l'omogeneità della microstruttura. Questi dati sono stati poi messi in relazione con gli aspetti più importanti del processo tecnologico adottato. La temperatura della billetta e del materiale deformato sono stati misurati con un pirometro ottico in differenti passi del processo. Una prima simulazione termica, basata sull'equazione di Fourier, è stata implementata al fine di comprendere la possibile influenza della trasmissione del calore e dell'omogeneità termica sulla qualità del prodotto finale. Rilevanti indicazioni sono state raccolte riguardo alle relazioni tra i parametri tecnologici adottati e la qualità dei risultati finali. Inoltre, sono state determinate alcune indicazioni riguarda l'entalpia sviluppata nel processo industriale di estrusione a caldo, che possono essere usate in un prossimo futuro per sviluppare più precise simulazioni numeriche del processo di estrusione.

INTRODUCTION

The use of the titanium alloys represents a good opportunity for several industrial sectors such as the automotive, aerospace, biomedical and energy ones. On the other hand, one of the most significant obstacles to the wide and fast diffusion of the titanium alloys is related to the difficulties about the performance of the plastic deformation processes. Actually, the titanium alloys show high flow stress even in the usual thermal range adopted in several hot deformation processes (950°C-1150°C) and this can be fundamentally explained on the basis of consideration about the lattice structures proper of the titanium alloys: the two possible phases which can be present, α-phase and β-phase, have a hcp and bcc lattice, respectively. These lattice structures are not suitable for a rapid and efficient plastic deformation, particularly in presence of the phase α, because the hcp lattice has only three independent slip systems involved in the plastic deformation and this number of systems is lower than five, that is defined as the lowest one which can grant the mutual adaptation among the lattices constituting the adjacent grains [1]. This crystal based plasticity consideration is the physical reason, which makes the plastic deformation of titanium alloys a process featured by a not simple management.

However, the difficulties belong to the technological field, too. The homogeneity of the heat distribution within the billet to be extruded, the thermal and chemical insulating, the decreasing of the friction realised by the used lubricating flux and the shape of the cross section are the most important ones [2,3].

The forward hot extrusion is a very suitable deformation process for the production of long and straight bar with constant cross section [3,4] and could be applied also to the most widely used titanium alloy: Ti-6Al-4V. The hot extrusion of Ti-6Al-4V (which is featured by a α–β structure at room temperature) has been experimentally studied also with the support of the simulation of the finite elements method [5,6,7,8,9]. These studies have improved the comprehension of the influence of the different factors involved in the deformation process, including also the lubrication and the heat transmission between the die and the extruded materials. Although a huge number of useful information can be found in these studies, they show some lacks, which affect the precision of the obtained results and their rapid application in the industrial situation. First of all, the information is often related to the case of a reduction from a starting round shape billet to the round cross section and not to the production of the special ones that are the more suitable for special applications. Moreover, the hypothesis of plane section of the material during the extrusion and the uncertainty of the data about the heat development during the deformation constitute two aspects, which can affect the precision of the simulation.

The present study is devoted to point out synthetic information about the critical metallurgical and technological items that have to be carefully controlled during the performance of the plastic deformation process, but also to reach some more reliable information about the heat development caused by the plastic deformation and the influence of the shape of the manufactured product. This aims to provide reliable data for a next and more precise simulation based on the Navier-Stokes' approach and to suggest to the industrial operators some practical and useful measures to be adopted for the hot extrusion of Ti-6Al-4V.

EXPERIMENT PROCEDURE

Twenty-four billets of Ti-6Al-4V (Chemical Composition in Table 1) featured by 100mm diameter and 500mm length have been extruded. The behaviour of the used alloy at high temperatures has been investigated on a tensile machine equipped with an electric induction device. For defining the relation between the yield stress and the strain rate the tensile test has been chosen instead of the compression test to avoid the problem of measurement generated by the friction between the Ti-6Al-4V samples and the machine equipment. The tests have been performed in nine different conditions of temperature and strain rate. The temperature has been set at 980°C, 1015°C, 1045°C and at each temperature three strain rates have been applied 0.01%s^{-1}, 0.02%s^{-1}, 0.03%s^{-1}. These tests have been performed in order to define the constant included in the hyperbolic-sine constitutive equation that has been demonstrated to be suitable for the description of the behaviour of metal at high temperatures [11].

The round billets have been extruded to obtain two types of product characterised by a different shape of the cross-section (fig. 1).

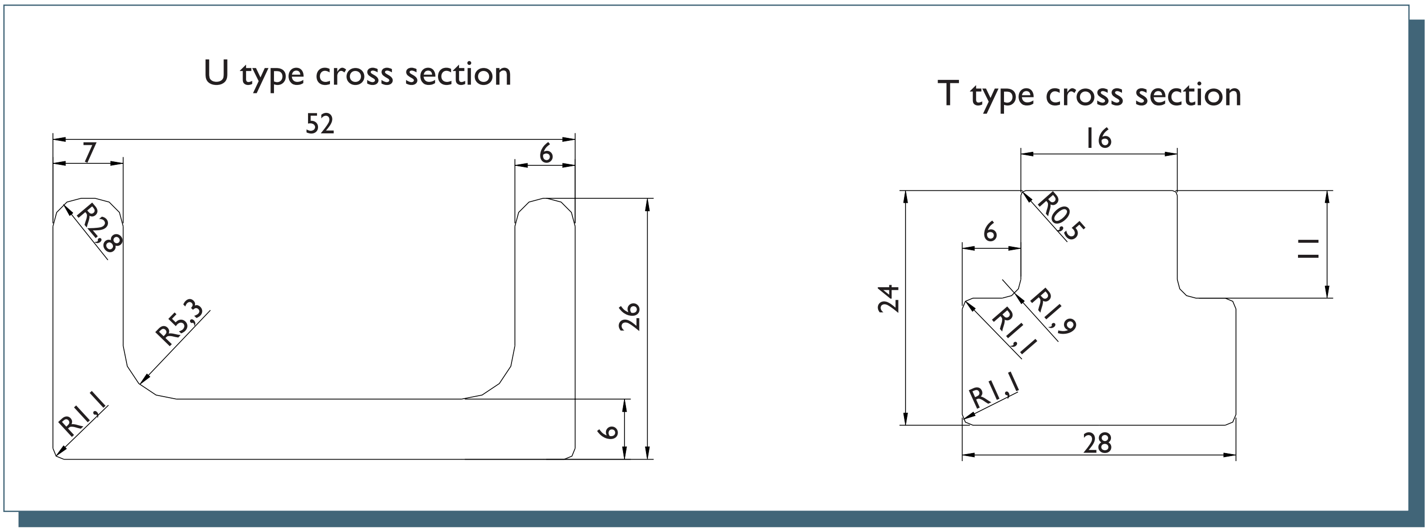

Fig. 1: Different shapes and dimensions (mm) of the cross sections of the extruded products: U-shape T-shape.

TABLE 1. CHEMICAL COMPOSITION (%WT) OF THE USED TI-6AL-4V.

C	Si	Al	V	Fe	Cu	Y	O	N	B	H
0.04	0.03	6.24	3.92	0.2	0.01	<.001	0.17	0.0065	-	0.0005

The extruded billets have been divided into two groups, each of which has been extruded in one of the adopted cross section shapes. The dies for the two cross-sections are 30mm long. The oxide flux used as lubricant is typical of the extrusion of high-alloyed steels (Table 2).

TABLE 2. CHEMICAL COMPOSITION (%WT) OF THE OXIDE FLUX USED AS LUBRICANT.

CaO	SiO2	Al2O3	TiO2	Na2O	KO	B2O3
11	56	21	2	1	1	8

A DTA (Differential Thermal Analysis) test has been performed to determine the β-transus temperature of the used Ti-6Al-4V.

The billets have been heated in an electric induction furnace and the temperature trend of the surface has been recorded by a digital pyrometer, which has been pointed within the furnace in a region at the middle of the billet height. The twelve billets have been divided into three groups featured by a different heating rate: 400°Cmin^{-1}, 230°Cmin^{-1} and 90°Cmin^{-1}. Two billets have been tested after heating at 980°C and the other two after heating at 1020°C. The temperature of each billet has been measured by a digital pyrometer 3 seconds before charging the chamber of the extrusion press. The temperature of the dies built in H13 steel is maintained at 350±10°C. The advancing velocity of the extrusion press has been set at 0.038 ms^{-1} for the U type cross-section and 0.034ms^{-1} for the other one. After 2s since the stop of the press two digital pyrometers have been used to measure the temperature of the surface, which is covered by the lubricating flux, and simultaneously two points within the cross sections have been measured. At the end of the extrusion operation the not deformed billet and the volume within the die have been extracted to evaluate quantitatively the effective volume of the material contained within the die (fig.2). The volume of the Ti-6Al-4V within the die has been determined by the immersion of the material portion itself in a container filled by water which is free to rise within a graduated column connected with the container itself.

All the samples have been examined by optical microscope to evaluate the possible presence of fractures. Three samples of the U type cross section and three of the T one have been cut from the tail of extruded profile to obtain the specimens, which would have undergone the characterisation of the microstructure by the metallographic etchings. By the Kroll's etching (1 ml HF, 3 ml HNO_3, water to 100 ml) the microstructure has been revealed out, while for the evaluation of the possible presence of the α-case (α-case: oxygen-, nitrogen-, or carbon-enriched, α-stabilized surface resulting from elevated temperature exposure [12]) an etching solution of 2ml HF-98ml H_2O has been used. Finally, the possible presence of hydrides has been evaluated by an etching featured by 2ml HF-10ml H_2NO_3-30ml lactic acid [13].

Fig. 2: An example of the not completely extruded material belonging to the last part of the billet.

RESULTS AND DISCUSSION

By a regressive computational method the values assumed by the characteristic constants and by the activation energy, which have to be used in the chosen hyperbolic sine constitutive equation have been evaluated:

$$\dot{\varepsilon} = A\left[\sinh(\alpha\sigma)\right]^{n}\exp(-Q/RT)$$

$$A = -13.65\varepsilon^{3} + 16.55\varepsilon^{2} + 0.85\varepsilon + 57.46$$

The values have been determined by averaging the results of the different tensile tests (Table 3).

TABLE 3. CHARACTERISTIC VALUES OF THE HYPERBOLIC-SINE CONSTITUTIVE EQUATION.

Q (kJmol^{-1})	α (MPa^{-1}s$^{-1/n}$)	n
380	0.053	6

The overall deformation rate within the die has been evaluated by averaging the deformation rate performed between the entry and the exit of each section:

$$v_i S_i = v_{i+1} S_{i+1}$$

$$\dot{\varepsilon}_i \approx \frac{v_i - v_{i+1}}{l}$$

The average value of the $\dot{\varepsilon}$ for the U type and T type cross section is evaluated to be the same, 17.0 s-1. On the other hand, the largest part of the deformation (87%) has been concentrated at the inlet of the die. From the experimental observations performed on the extruded samples belonging to the last part of the billet, this zone has been estimated to be 1mm length and is featured by a deformation rate of 74s-1. This last value has to be considered in order to evaluate the enthalpy development. The average flow stresses of the section during the deformation in the die, computed on the basis of the constitutive equation, assume the average value of 125MPa and a standard deviation of 5.2MPa for the U type cross section and 131MPa and 8.3MPa, respectively, for the T type cross section.
The heat developed by the hot extrusion process has been computed on the basis of the temperature difference measured on the billet and on the extruded product. Actually, the developed enthalpy is related to the thermal increase by:

$$\Delta H = mc_p\Delta T$$

where on the basis of the volume measurements performed on the material contained in the die the mass *m* assumes the value of 0.0273kg for the U type cross section and 0.0267kg for the T type one, with a density of 4510kgm^{-3}. The average thermal difference measured for the U type cross section is 148°C while that for T type one is 134°C. Provided the thermal difference produced by each extrusion operation, the enthalpy has been computed and is related to the plastic deformation working parameters by [13]:

$$\Delta H = \xi\sigma\,\dot{\varepsilon}\,\Delta t\Delta V$$

By this equation the average value for adimensional constant ξ has been computed to assume a value of 0.94 and a standard deviation of 0.03, which is also coherent with the data computed for other materials [14,15,16]. This conclusion based on experimental evidences can make the next simulations more precise because it clarifies, on the basis of experimental results, the relation between the developed enthalpy, the flow stress and the deformation rate.
The observations about the quality of the final product have pointed out that seven extruded product are interested by inner cracks which make the final products unacceptable (Table 4).

Table 4. Presence of cracks in the extruded products.

Cross section	Heating temperature (°C)	Heating rate (°C/min)	Extruded samples	Sample with cracks
U	980	400	2	2
		230	2	2
		90	2	2
	1020	400	2	1
		230	2	0
		90	2	0
T	980	400	2	0
		230	2	0
		90	2	0
	1020	400	2	0
		230	2	0
		90	2	0

The cracks have been concentrated in the U type cross-section and in correspondence of the highest heating rate and the lowest temperature of extrusion.
However, the detrimental effect of the applied highest heating rate seems to be excluded on the basis of the simulation which has been developed using as boundary condition the temperature measured by the thermal measurements of the billet within the induction furnace. The simulations have been performed by a finite difference method for describing the heat transmission in radial coordinates [17] using as a reliable value for the thermal conductivity 11Wm^{-1}K^{-1}[12]. The temperature between the surface and the core of the billet can be calculated at the different temperatures by the following relations, which have

been computed by interpolating the results of the performed thermal simulation:

$$T(z) = -1085.4z^2 + 11.87z + T_{surface} \qquad 400°C/min$$

$$T(z) = -624.08z^2 + 6.77z + T_{surface} \qquad 230°C/min$$

$$T(z) = -244.2z^2 + 2.65z + T_{surface} \qquad 90°C/min$$

The heat difference between the core and the surface is not so significant to produce problems during the hot extrusion process. Only heating rate of 2000-2500°C/min can produce a significant thermal difference between the surface and the core of the billet, but this heating rate is not usual in the industrial extrusion plant. It is evident enough that the only influence of such a small thermal difference cannot explain the failure phenomena and the scattering observed in the heating on the surface of the extruded material.

The DTA measurement has allowed to determine the β transus temperature, which is in a range of 1003-1012°C. So, it appears clearly that the extrusion processes, which begin under the β-transus temperature, are more critical and the coexistence of α-phase and β–phase has to be avoided. Moreover, at higher extrusion temperature the used lubrificant softens sufficiently to possess liquid characteristics: at strain rates higher than 15 s^{-1}, like the ones considered in the present investigation, there is not sufficient time for the lubrificant to be squeezed out, resulting in favourable lubrification conditions [3]. On the other hand, these ones don't appear the only determinant factors of influence, because none of the T type cross sections show the development of any crack. A relation among the failure events and the surface temperature of the extruded product at the exit of the die can be recognised. The temperature increase of the surface seems to indicate that there is a relation between the observed failure events and the thermal heating of the lubricating flux (Table 5).

Table 5. Heat temperature of the surface of the extruded product at the exit of the die.

Cross section	Heating temperature (°C)	Heating rate (°C/min)	Extruded samples
U	980	400	1038
		230	1030
		90	1027
	1020	400	1045
		230	989
		90	963
T	980	400	984
		230	956
		90	937
	1020	400	898
		230	888
		90	847

The highest levels of heating, have been observed when the cracks have taken place: the extrusion of the U type cross section seems to be more critical than the T type one. The ratio between the final contour perimeter of the cross section and its area is 0.35m^{-1} for the U type cross section and 0.17m^{-1} for the T type one. This means that the U type cross section is composed by thinner sections than the T type and offers a larger area of interaction between the die and the extruded material.

The local strain rate of the U type cross section is greater than the one of the T type and it is proved also by the heat globally developed during the extrusion process as it has been witnessed by the average greater thermal increase. This implies an increase in the flow stress of the material and then an increase in the friction between the die and the extruded product. The great thermal increase of the surface can only be due to the increase of the friction pressure and the work dissipated through this mechanism. The high thermal increase concentrated on the surface exalts the mechanical difference between the mechanical features of the surface itself and the core of the extruded product. This can produce a difference in the flow velocity of the material within the die. It is worth noting that the fracture events have been observed in the horizontal and vertical long side of the U section (fig.3, fig 4). The design of the die causes a larger strain rate on the portion of material, which is pulled from the entry section towards the thin vertical and horizontal side zones of the cross section. These ones represent the sides of the section featured by a long perimeter, the overall exchanged force is greater, so the friction force is larger and larger is the heat dissipated and concentrated on the surface of this region. The possible presence of some fractions of α-phase, which is more difficult to be plastically formed, can make the situation worse, by increasing the difference of the flow velocity between the surface zones, which have a temperature over the β transus, and the core zone, which presents the α-phase.

A quantitative description of this phenomenon would be improved by the use of Navier-Stokes' equations to simulate the flow of the extruded material which permits to evaluate the different acceleration of the material within the die.

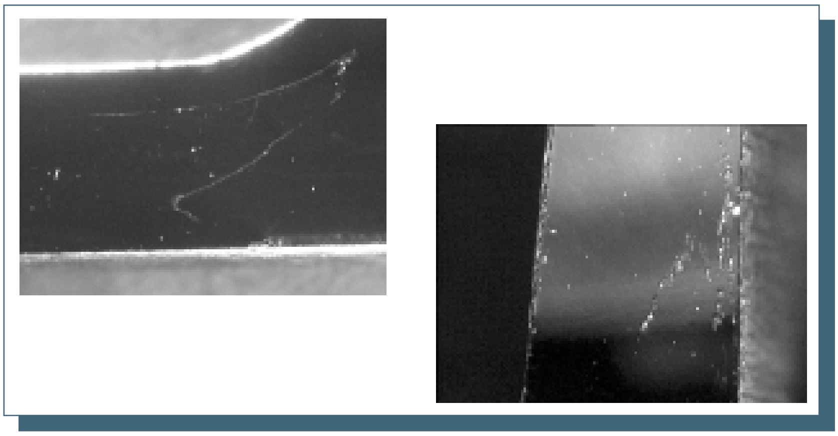

Fig. 4: Some internal defects found in failed samples.

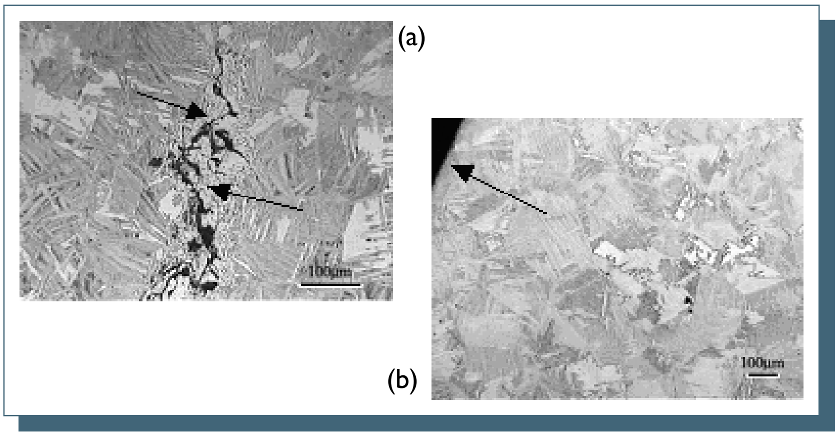

Fig. 5: Only near the crack there is the possibility to form α-case (A), at surface no α-case is observed (B).

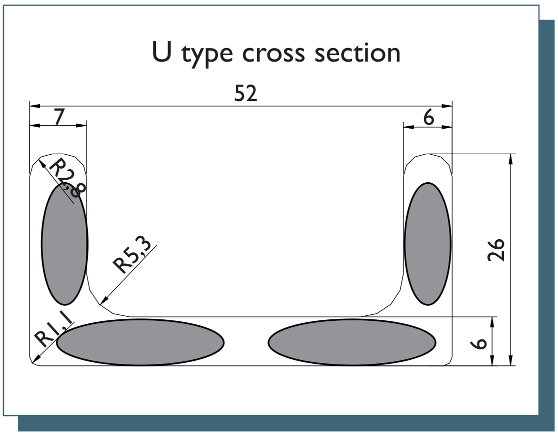

Fig. 3: The grey zone represents the location of the observed fractures on the failed samples.

The used glass lubricant flux seems to have performed a good chemical insulating action between the extruded Ti-6Al-4V and the surrounding atmosphere, because the α-case has not been revealed on the surface of the extruded materials. The molten glass during the hot extrusion has always formed a uniform distributed coating which shielded the titanium alloy. The only presence of the α-case has been observed near the cracks where the titanium alloy cannot be protected by the interaction with the gases contained in the atmosphere, which have been absorbed at high temperature and causes the presence of the α-case (fig.5). In all the observed samples no surface cracks due to poor lubrication have been found.

In the well performed extruded materials the final microstructures have shown a homogeneity also between the boundaries and the core regions: α at prior β grain boundaries and transformed β containing acicular α were observed (fig.6 and 7). No hydrides have been found in any sample.

Fig. 6: A typical observed microstructure in T and U cross section: α at prior β grain boundaries and transformed β containing acicular α.

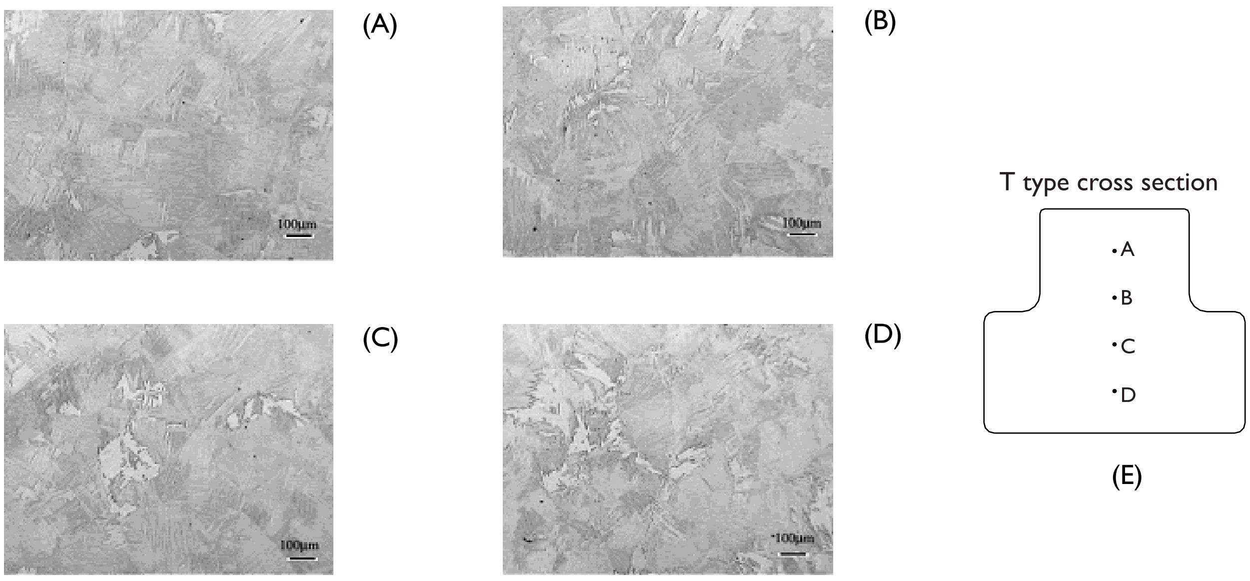

Fig. 7: The microstructure appears homogeneous in the entire cross section (see (E)).

CONCLUSIONS

The present study has been devoted to the definition of fundamentals of hot extrusion of Ti-6Al-4V:

- the characteristic constants of the sine-hyperbolic constitutive equation of the Ti-6Al-4V by tensile tests performed at high temperatures and at different strain rates;
- the characteristic constant of the relation among the developed enthalpy, the applied stress and the strain rate has been determined to be 0.93 and it is coherent with that characterising other industrial alloys;
- the temperature of hot plastic deformation should be slightly higher (to avoid the formation of hydrates and the formation of "pipe" defects) than the β transus which for the used titanium alloy has been measured to be in a range of 1003-1012°C;
- the higher is the ratio between the perimeter and the area of the extruded section, the thinner is the thickness of the parts composing the cross section and the local strain rate applied to regions of the extruded material that can produce a higher heat concentration on the surface of the material;
- to avoid too large gradient in the flow velocity between the boundary and the core region (especially when the cross sections with high ratio between perimeter and area have been extruded) a temperature under the β transus has to be accurately avoided;
- the large heat concentrated on the surface can be promoted by high local strain rate and this can generate high gradient of flow velocity between the core and the boundary of the product during the extrusion;
- in all the analysed samples a good uniformity of the final microstructure has been shown;
- the presence of the α-case has been limited to the surface of the observed cracks which can absorb the atmosphere gases, while on the surface of the extruded product the used lubricant flux seems to have operated a good chemical shielding action.

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LIST OF SYMBOLS

α	characteristic constant of the constitutive equation (MPa^{-1}s^{-n})
ξ	characteristic constant of the material for the enthalpy development
ΔT	thermal difference between the entry temperature of the billet and that of the just extruded material (°C)
ΔV	volume of the material contained within the die (mm^3)
Δt	time spent by the extruded material to go through the die (s)
ΔH	enthalpy developed by the plastic deformation (J)
$\dot{\varepsilon}$	overall and averaged strain rate realised by the die (s^{-1})
$\dot{\varepsilon}_i$	the strain rate realised in the die (s^{-1})
ε	the deformation
σ	the flow stress (MPa)
A	strain dependent parameter
c_p	specific heat of the titanium alloy (522Jkg^{-1})
l	length of the die (m)
m	mass of the material contained in the die (kg)
min	minute (60s)
n	characteristic constant of the constitutive equation
Q	the activation energy of the process (kJmol^{-1})
R	perfect gas constant (8.31JK^{-1}mol^{-1})
S_i	area of the section just before the entry of the die (m^2)
S_{i+1}	area of the section at the exit of the die (m^2)
T	absolute temperature (K)
$T(z)$	temperature of a point on the radius of the billet positioned at a distance *z* from the surface(°C)
$T_{surface}$	instantaneous temperature of the billet surface (°C)
t	time spent by the material from the entry section of a discretized volume to the exit one (s)
v_i	velocity of the material at the section just before the entry into the die (ms^{-1})
v_{i+1}	velocity of the material at the exit of the die (ms^{-1})
z	coordinate defining the position of a point from the surface to the centre of the billet (m)

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