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ORIGINAL RESEARCH

Mechanical properties and microstructure of Inconel 625 cylinders used in aerospace industry subjected to flow forming with laser and standard heat treatment

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Abstract

The aim of the current study was to obtain a hollow axisymmetric cylinder that can be used in the aerospace industry. Mechanical properties and microstructure of Inconel 625 after cold forming and subsequent heat treatment were analyzed. The results show that due to cold forming process the strength of the workpiece drastically increased (up to 1600 MPa) at the cost of plasticity. To obtain good combination of mechanical properties, the material was additionally using two heating methods: laser and resistance furnace annealing. The main purpose of the experiment was to prepare manufacturing guidelines for aerospace industry requirements. The results obtained in the experiment enabled to obtain a part that meets the requirements of the industry. Additionally, laser and furnace heat treatment were compared as well as their mechanical properties and microstructure of the material.

Keywords Laser assisted metal spinning · Inconel 625 · Heat treatment · Formability · Metal forming · Gas turbine

Introduction

Flow and shear forming are versatile methods used to produce hollow axisymmetric parts. The principles of the forming process are easy to understand and are associated into simple mechanics. A rotating sheet of metal (the workpiece) is placed on rotating die – mandrel. The next step involves plastic deformation by rollers rotating around their own axis. Depending on the used tooling their number may vary from 1 to 3 sometimes more according to Music et al. [1] (Fig. 1). Both metalworking techniques are still developed and improved. In comparison to the techniques used in the middle of the XX century, stiffer and more durable tooling are now used. Additional computer numerical control enables greater accuracy and repeatability of the process parameters according to Wong [2]. Furthermore, there are multiple methods that enhance the formability of the material mainly through modification of tooling. The modification of flow and shear encountered in the literature are laser heating proposed by Klocke and Wehremeister [3], stiffer and more durable tooling and non-axisymmetric forming proposed by Xia et al. [4]. Thanks to those improvements the applicability of the two metalworking techniques is still growing and evolving.

There are numerous advantages of both method that make them competitive to other stamping methods e.g. high strains obtainable in one process, universal tooling and above average mechanical properties. The manufactured parts have good strength and acceptable plasticity thanks to the favorable mostly compressive stress state which prevents localization of deformation and cracking. There are few research papers that studied this phenomenon in terms of mechanical behavior of the workpiece after the forming process due to the circular shape of the element. Lee et al. [5] in their work studied the mechanical properties of Maraging steel. They obtained ultimate tensile strength (UTS) in the range from 1145 MPa up to 1960 MPa with an additional aging heat treatment. This is due to structure refinement and additional enhanced precipitation hardening caused by severe plastic deformation. Abedini et al. [6] in his results show that thanks to flow forming it is possible

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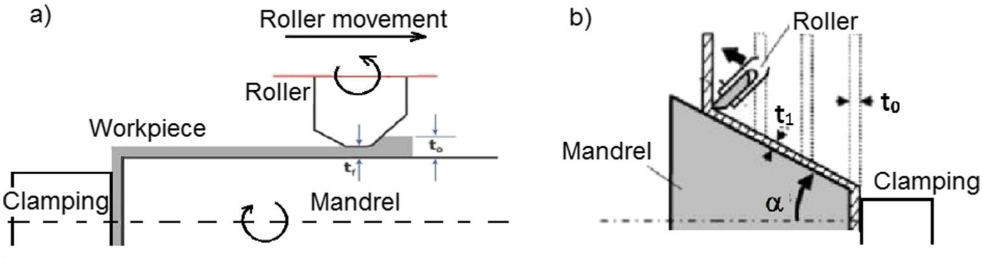


Fig. 1 Two metalworking techniques (**a**) flow and (**b**) shear forming

to enhance the strength of polymers. Furthermore, according to Cao et al. [7] even hard to deform materials with limited deformation capabilities like magnesium, can be formed enhancing their tensile properties additionally.

Inconel 625 (Table 1) is a nickel based superalloy with superior corrosion and temperature resistance. Tt has very high strength and good ductility due to niobium and molybdenum solution strengthening. The effect of the precipitates is relatively small, although subsequent heat treatment causes them to appear in the material. Reinforcing phases at temperatures above 700 °C are γ “and at a later stage of the heat treatment phase δ. The standard heat treatment is conducted in 982 °C for 8 h according to AMS 5599. Solid solution of γ equiaxed grains was observed. However, it should be noted that the strain induced in the material can have a significant effect on the microstructure changes during annealing according to Wei et al. [8]. High density defect sites can be nucleation sites of new grains and precipitates. Furthermore, time and temperature can change the microstructure and, therefore, mechanical properties of the material.

The aim of the conducted experiments was to analyze the effect of heating conditions on the mechanical properties and microstructure of Inconel 625 after flow forming process. Two types of annealing were compared: the traditional furnace heating which is more time consuming but more consistent and widely used, and laser heating which is a dynamic and fast process. In addition, the second heating method was conducted in industrial conditions using a laser assisted flow forming machine. The overall purpose was to eliminate interoperational annealing during flow forming of axisymmetric parts manufacturing. Additional goal, with the cooperation with Pratt & Whitney Rzeszow was set to obtain a large cylindrical part which has appropriate geometry. To do this the first stage of the experiment was conducted on relatively small preforms to acquire appropriate data for large scale elements testing.

Experimental

The studied material was Inconel 625 obtained according to AMS 5599. Initial small preforms (Fig. 2) were obtained by deep drawing. To homogenize the microstructure a standard heat treatment was conducted. The next step involved forming of the workpieces using a Leifeld prototype SFC 800 V500 machine with a laser module enabling circumferential heating of the workpiece (Fig. 3). It is worth noting that the process configuration is not standard to flow forming due to two major reasons. First, the overall objective of the conducted project was to combine three processes conventional metal spinning, flow forming and shear spinning. It was necessary to reach a compromise between the tool geometry which is different between individual metal spinning process. Furthermore, due to high temperature special heat resistant materials were used which significantly increased the cost of additional tooling. Secondly, the use of a laser heating caused an issue with the heat dissipation on the rollers. It was necessary to reduce the contact zone between the workpiece and the roller to increase the efficiency of heating.

The metal working process was conducted with different thickness reduction to determine the main forming limitation of the method. To obtain the required thickness, the gap between the roller and the mandrel was kept constant and changed in individual technological tests. The deformation was performed in one movement of the tool. What is worth noting the samples used in the first stage were relatively small in diameter and height (Fig. 2) and were pre-processed by pressing which significantly reduced the costs of their manufacturing. In the second stage the pre-form cylinder had much higher dimension (c.a. 50 cm diameter and 40 cm height) which greatly increased the price of prefabricate although the geometry reassembled a gas turbine. The aim of the tests was to obtain geometry of the workpiece that can be

Table 1 Chemical composition of Inconel 625

	Ni Chemical composition [wt.%]	Cr	Fe	Nb/Ta	Mo	Ti	Al	Co	Mn	C	Si	P	S
Max	–	23.00	5.0	3.15	10.00	0.4	0.4	1.00	0.5	0.1	0.5	0.015	0.015
Min	58.00	20.00	–	4.15	8.00	–	–	–	–	–	–	–	–

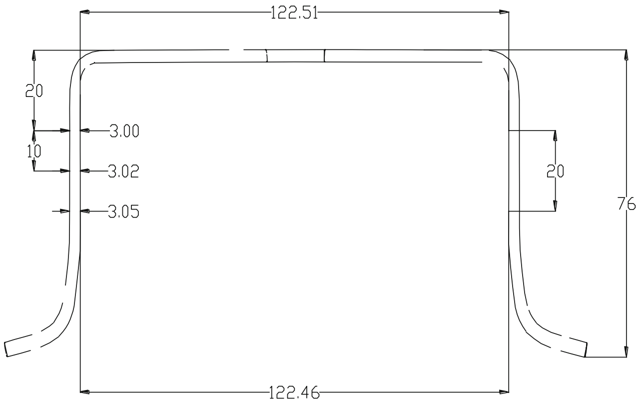


Fig. 2 The geometry of the prefabricate

used in a real part of a jet turbine case. It is worth noting that this engine is widely used and normally obtained by multiple pressing and furnace heating.

The prefabricate used to obtain the large sized cylinder was machined from a sheet of metal ordered directly from the supplier according to AMS 5599. Hydraulic press was next used to achieve the initial shape of the cylinder. After this process a standard heat treatment was conducted. Material with properties similar to the initial state, suitable for further metal working was obtained. The next step involved shear and flow forming (Figs. 3 and 4). Additionally, in the lower flange where the material was susceptible to cracking laser heating was used to increase the formability by decreasing the strain hardening effect caused by deformation (Fig. 4). The temperature measured at the surface was in the range of sub solvus hot forging [9].

The process configuration was not standard due to three factors. The overall aim was to combine three different processes conventional metal spinning, shear and flow forming in one complex forming process. As a result, the process configuration was modified towards conventional spinning operation. Furthermore, the cost of tooling was very high due to laser heat treatment and the necessity to use special heat resistant materials. Additionally, the time of rearming of the machine was significant. Ultimately a compromise was established to obtain the required geometry in a sufficient short time.

Tensile tests were carried out using mini-samples with gauge lengths of 10 mm. The experiment was conducted using a Zwick/Roell 005 machine at an initial strain rate of 10^{-3} 1/s. The tests were done at room temperature. Optical non-contact displacement measurement by Digital Image Correlation technique for precise elongation measurement was used according with Molak et al. [10] procedures. The cylinders were tested in the radial direction. The dimension of the mini-samples used in the experiment is shown in Fig. 5. The samples were extracted from the middle part of the cylinders. Their small dimensions enabled to decrease the effect of the cylinder curvature. Additionally, grinding with a sand paper was used to obtain parallel surfaces. Ultimately the samples in the experiment broke mostly in the middle section which proves that the tests were properly prepared in terms of uniaxial tensile analysis.

The tensile samples were extracted from the middle part of the cylinders (Fig. 5). The cylinders were tested in the radial direction. The samples cut from cylinders had a small curvature. To obtain reliable results and reduce the impact of the curved geometry, mini-samples with gauge lengths of 10 mm were used. Furthermore, the samples were grinned on abresive paper to remove any bulges. The dimension of the mini-samples used in the experiment is shown in Fig. 5. Tensile tests were carried out a Zwick/Roell 005 machine at an initial strain rate of 10–3 1/s. The tests were performed at room temperature. The experiment was conducted using for precise elongation measurement optical non-contact displacement measurement by Digital Image Correlation 2D technique was used according with Molak et al. [10, 11] procedures. The stress-strain curves did not suggest any shape/curvature influence. Moreover, the samples cracked mostly in the middle of length section which is correct in terms of uniaxial tensile test standards.

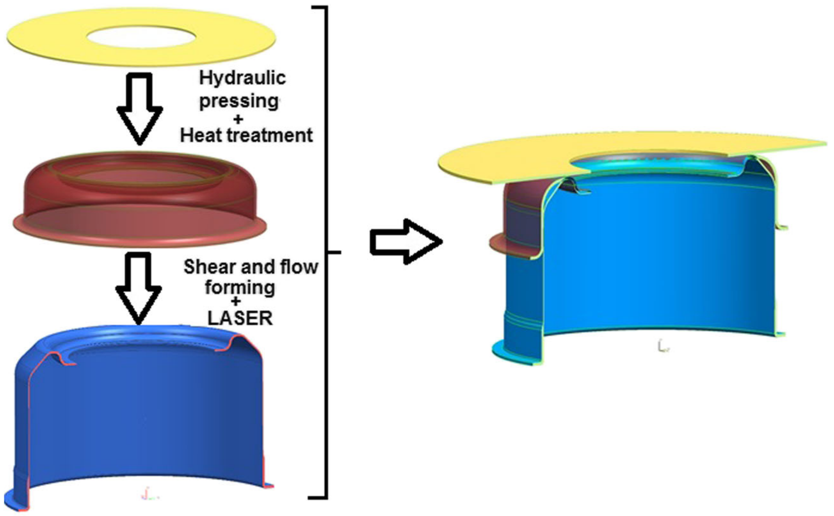


Fig. 3 The schematics of the metal forming processes

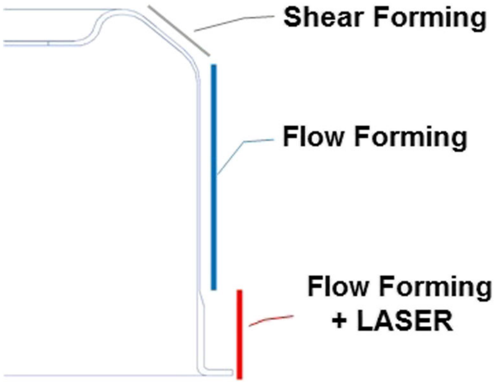


Fig. 4 The schematics of flow and shear forming processes

For optical microscope (OM) the selected material specimens were elektroetched using a Struers machine and 15 mL HCl, 10 mL glicerol and 5 mL nitric acid etchant.

The residual stresses were measured using the Bruker D8 Discover X-ray diffractometer with a point beam collimated to approximately 1 mm Cr $K_{\alpha 1}$ (2.29 Å) radiation. Measurements were provided by $\sin^2 \Psi$ method which is considered as a non-destructive method among many stress determination methods. X-ray diffraction residual stress measurement uses the distance between crystallographic planes d as a strain gauge. The deformations cause changes in the d spacing of the lattice planes from their stress-free value to a new value that corresponds to the magnitude of the residual stress. Because of Poisson's ratio effect, if a tensile stress is applied, the lattice spacing will increase for planes perpendicular to the stress direction, and decrease for planes parallel to the stress direction. The diffraction angle (2Θ) is measured experimentally and then plotted versus sin2 Ψ (Ψ is the specimen tilt angle). The residual stress measurement was performed after the component was cut by WEDM method which did not induce any additional stresses. Nonetheless after the extraction of the sample elastic deformation was reduced. However, it should be noted that the XRD analyses mainly the residual stress at the atomic level which is correlated with lattice distortion which is induced mostly as a result of plastic deformation. Therefore, the influence of the cutting process on the residual stress is negligible and it was not considered at the present work. The XRD results are qualitative rather than quantitative due to various factors concerning the cylinder geometry, heterogeneity of deformation, texture effects and the impact of the flow forming parameters. Nonetheless the values presented in the text are sufficient to supplement the mechanical results and structure changes that occurred after metal forming process. A more detailed analysis concerning residual stress analysis in the flow formed cylinders was conducted by Tsivoulas et al. [12].

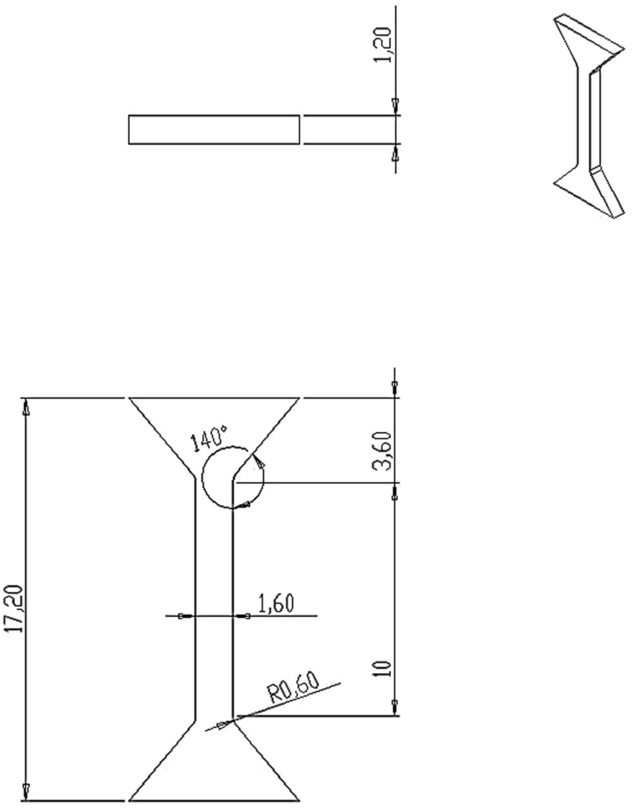


Fig. 5 The dimensions of the mini-samples used in the experiment

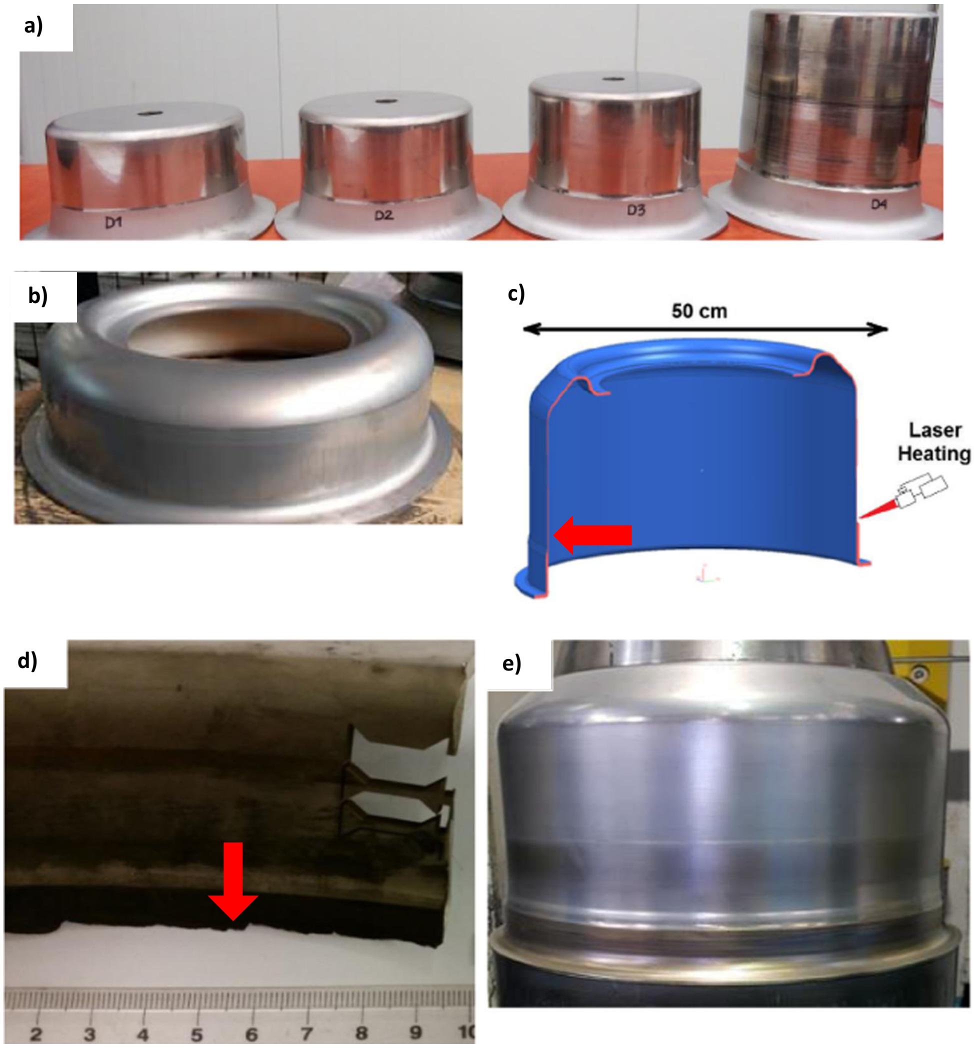
Additional transmission electron microscopy (TEM) characterization was carried out The samples (100 μm thick disks with a diameter of 3 mm) were cut from heat treated sheets of Inconel 625 using wire electro-discharge machining (WEDM). The foils were next electropolished using A8 electrolyte provided by Struers in a similar manner. The observations were done using a JEOL JEM 1200 EX 2 microscope, with an accelerating voltage of 120 kV.

The results

Technological tests

The technological tests were conducted first for small cylinders (Fig. 6a). Four degrees of deformation were induced in the cylinders in one roller movement along the radial direction (Table 1). No surface defects were observed in the material even at cold forming process after 70% cross-section reduction. It is worth noting that the set deformation (based on the gap between the roller and the mandrel) was lower than obtained which is a result of a springback effect which is dependent on process parameters and the stiffness of the machine. Based on the results from the first stage (small cylinders) the necessary corrections were made to obtain the set the required wall dimension. The experiment was conducted to obtain up to 70% thickness reduction. The large cylindrical element was obtained from a preform shown in (Fig. 6b) Unfortunately later after the cold forming process cracks were observed in the lower flange of the cylinders (Fig. 6c and d). To overcome

Fig. 6 Flow forming (**a**) initial tests for small cylinders (**b**) preform for the large cylinder, (**c**) cracking localization, (**d**) separation surface, (**e**) final product after laser heat treatment



this obstacle, local laser heating was used to avoid intermediate annealing. It is worth noting that the roller can move in sync with the rollers. However simultaneous forming and laser heating causes clamping due to overlapping of thermal contraction and plastic deformation. Overall it is highly advisable to separate both processes especially when the temperature gradients are high like in the present case. First, carry out the metal forming process and then use the laser heat treatment to reduce the strain hardening effect and increase the formability of the material. Additionally, for comparison reasons conventional heat treatment was conducted for the cylinder after deformation. The final workpiece (Fig. 6e) met the geometrical requirements of Pratt & Whitney Rzeszow and had no flaws that excluded the element from further machining.

Mini-tensile tests

The mechanical tests were done on samples cut from the cylinders obtained in the technological tests. The results showed that the high strength can be obtained after flow forming although at a cost of elongation (Table 2). Thickness reduction was followed with an increase of Yield (YS) and Ultimate Tensile Strength (UTS) of the formed element. In terms of values after achieving total strain of 67% the strength of the material rose to almost 1600 MPa. Unfortunately, the process involved a significant decrease of elongation. Furthermore, from the metal forming perspective it was necessary to obtain a sufficient level of plasticity to prevent the element from uncontrolled brittle cracking. The standard process involved heating in a furnace for a longer period (several hours) in a temperature range 900 °C – 1000°. To shorten this time laser heating was used. The experiment focused on the laser assisted flow-forming process. The tensile specimens were taken from heat affected zone and the cold formed surface. Furthermore, samples after a standard heat treatment was used as a reference. Comparing those results (Fig. 7) (Table 3), it should be noted that the UTS and YS of the laser heated specimens was lower than in the initial state. The elongation

Table 2 Initial tests – mechanical properties of the obtained cylinders

No.	Thickness [mm]		Total strain [%]		Mechanical properties		
	Set thickness	Obtained thickness	Set strain	Obtained strain	YS [MPa]	UTS [MPa]	Elongation at break [%]
1.	1.47	**1.74**	25	**11**	919	1066	17
2.	1.18	**1.52**	40	**22**	1086	1218	9
3.	0.78	**1.22**	60	**38**	1197	1352	7
4.	0.2	**0.65**	90	**67**	1319	1578	6

The bold entries emphasize the difference between the set value that is obtained and the set value

remains at a similar level which is beneficial in terms of metalworking process. Nonetheless it was done at a cost of strength of the final workpiece. However theoretically it is still possible to optimize these properties by using different laser power and heating time.

Optical microscopy

The microstructure of the specimens used in the experiment are shown in (Fig. 8) The material in the initial state had equiaxed grains (Fig. 8a). After the flow forming process long elongated bands of material can be seen (Fig. 8b). It is worth noting that their thickness decreases near the surface of sample which is a result of higher deformation. The microstructure of the specimen after annealing at first glance was similar to the initial state. Some annealing twins can be seen although the difference between grain sizes were very low (Fig. 8c). On the other hand, the samples that were subjected to laser heating had a higher spread of grain sizes (from 5 μm to 15 μm). This changed with the distance from the surface. Higher degree of deformation promoted more intense recrystallization effect which can be seen in Fig. 8d.

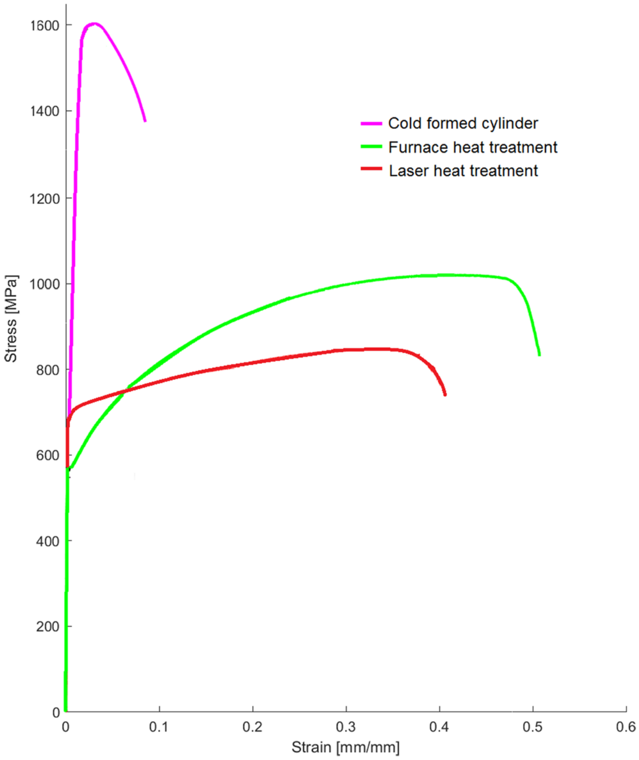


Fig. 7 Tensile tests of large cylinders

Residual stress analysis

Residual stress analysis (Fig. 9) was conducted to quantify the deformation that was induced during the cold forming process and laser heat treatment of the large cylinder. The values were extremely high in comparison to the initial state of the material and were a result of severe deformation especially near the surface area (2). The residual stress was lower in the inner surface which suggests a gradient of deformation along the thickness of the formed element. What is worth noting that the heated region (1) had relatively low stress which proved beneficial in terms of further plastic deformation. The high values of residual stress in this case are indirectly proportional to strain hardening which is commonly known to reduce the formability. By laser annealing the effect is significantly reduced due to dislocation rearrangement and recrystallization. This overall is beneficial in terms of further plastic deformation of the material.

TEM observations

To understand the changes that occurred in the material after the heat treatment TEM observations were done. Three materials were tested mainly in terms of precipitations and defect concentration. In the initial state, Inconel 625 had only few carbides visible in the matrix (Fig. 10a). Furthermore, the concentration of the dislocations was very low which Is typical in case of fully annealed material. After the cold forming the material was severely deformed due to induced strain. The concentration of dislocation was very dense creating abundant forest dislocations throughout the whole volume. An interesting feature are multiple shear bands that have a width in the range of 20–100 nm (Fig. 10b). Moreover, the material after the laser heating had also a large concentration of dislocations

Table 3 Tests conducted on the final large-scale cylinder

No	Sample	Thickness [mm]		Total strain [%]		Mechanical properties		
		Set thickness	Obtained thickness	Set strain	Obtained strain	UTS [MPa]	YS [MPa]	Elongation at break [%]
1	Cold formed Inconel 625 (large cylinder)	1.03	**1.4**	77	**70**	1610	1420	7
2	Laser heated fragment Inconel 625	1.84	**2.33**	59	**48**	792	682	41
3	Conventionally furnace heated Inconel 625	1.8	**2.2**	90	**67**	982	572	52

The bold entries emphasize the difference between the set value that is obtained and the set value

although they were less dense and better arranged forming organized structures (Fig. 10c). The dislocations grouped around selected crystallographic directions. Few participates were seen in the matrix that were cut by multiple linear defects (Fig. 10d) On the other hand, the furnace heat treated sample had equiaxed grains with low density of dislocations typical for materials after recrystallization (Fig. 10e). Neththeless, numerous particles can be seen in the microstructure that are anchored by dislocation clusters. This increased the overall strength due to precipitations hardening of the material in comparison to the initial state material.

Discussion

The aim of the current research was to obtain a large hollow cylindrical part from Inconel 625 which has acceptable mechanical properties. The tests in the first stage have confirmed the good formability of the workpiece, although as it turned out in the second stage, large scale elements are more demanding due to higher stress concentration in the volume of the element and cracking. This problem was overcome after heating the material with a laser built-in the machine along the circumference of the cylinder. After heating, the material it

Fig. 8 Inconel 625 (**a**) in the initial state, (**b**) after deformation (total strain 70%), (**c**) furnace and (**d**) laser heat treatment

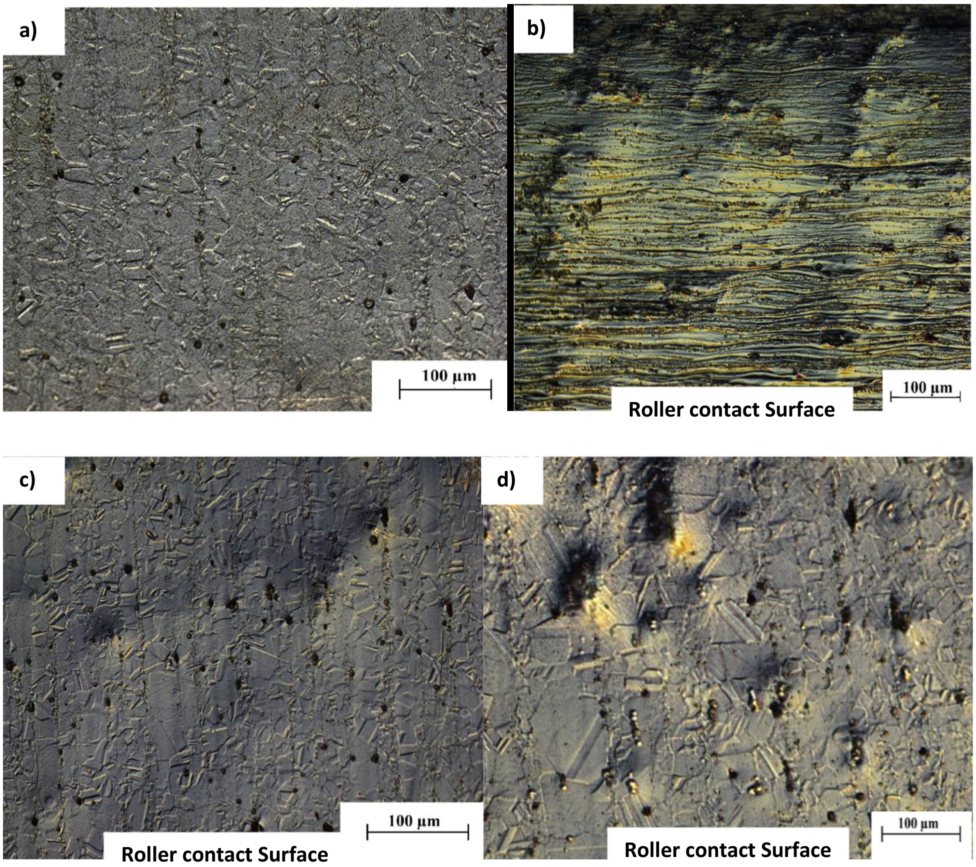
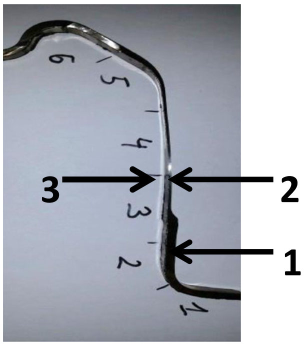


Fig. 9 Residual stress of the laser assisted flow formed element

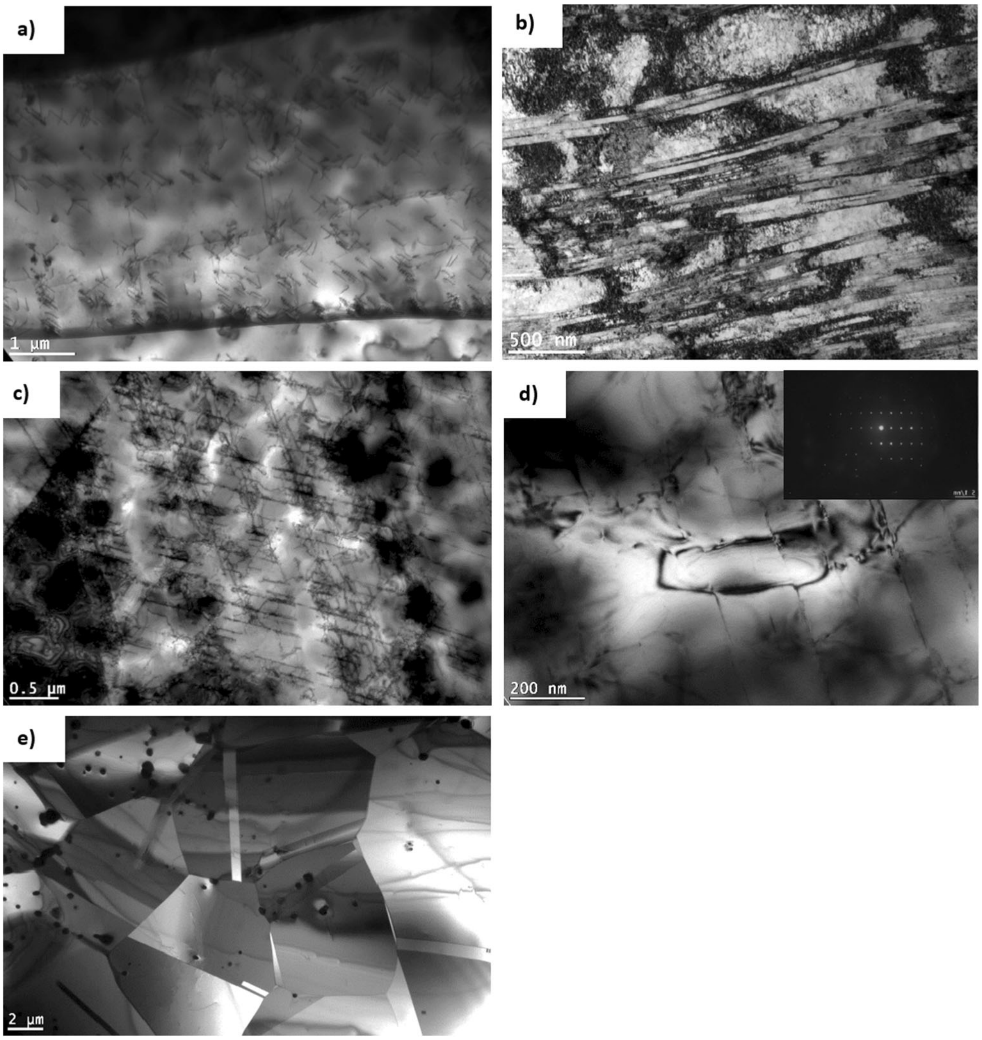


No.	Residual Stress [MPa]
1	-113 ± 19
2	-1294 ± 32
3	-458 ± 29

deformed easily without any cracks, which were seen in the cold formed cylinders, due to excess residual stress. It is worth noting that the cracks were seen only near the flange. By local heat treatment the overall time of the manufacturing process was shortened in comparison to normal furnace treatment. Furthermore, the microstructure was changed only locally which altered only a small volume of the material.

Several differences were observed between the individual samples due to different heat treatments. In case of the cold process high residual stress near the surface contact area will lead to cracking and low plasticity. This overall reduces the formability of the material. To overcome those drawbacks heat treatment must be used. Two heating processes were used and compared. The results showed that, from the point of sheet

Fig. 10 TEM observations (**a**) the initial state, (**b**) micro-shear bands after deformation (70%), (**c**) organized dislocation structure after laser heating (**d**) multiple shearing of precipitate (**e**) structure after furnace annealing



metal working technique laser heating, has the most benefits. Among the most important: lower strength, higher elongation, shorter metal working time. The problems that occurred in the experiment were related to inhomogeneity due to two independent factors which additionally overlap each other. The deformation in the flow forming process is the highest near the surface due to intense contact with the roller. The current results verified this observation. Deformation bands became thinner near the surface. Furthermore, the concentration of dislocation was very high. These factors reduce the recrystallization temperature. The heat spreads from the surface overheating the element. As a result, slightly higher grain sizes were observed which overall decrease the strength of the material. Similar results concerning the recrystallization phenomenon was described by Li et al. [13] where large spread of grain sizes (from 5 to 24 μm) were observed depending on the annealing parameters. On the other hand, comparing the current results to the material properties provided by the suplier [14]. for high temperature annealing 1200 °C similar strength can be observed (about 800 MPa) for the material obtained in the current experiment.

Comparing the results of standard heat with laser treatment, various factors should be considered. First, prolonged time will lead to evenly distributed precipitations creation and a more homogenous microstructure due to long term ordering and dissipation of redundant dislocation at the grain boundaries. Contrarily short annealing times will lead to favored nucleation sites and uncontrolled growth of some grains with excess of energy caused by plastic deformation according to Tehovnik [15]. Another factor that need to be addressed is temperature gradients that is relatively low during the furnace annealing in comparison to laser treatment. Rapidly heated and cooled material is subjected to thermal shocks and stresses which are introduced due to linear expansion near the surface area. This affects the microstructure on the sub-microlevel making a difference in the dislocation density and their organization. On the other hand, the precipitates are not as common as in the initial state decreasing overall the strength of the material. On the contrary the deformed material had much different microstructure than the previously described. It is similar to materials after severe plastic deformation described by Valiev [16]. Dislocation walls and shearing bands were dominant throughout the tested volume of the material. This translates into high strength of the deformed material. After annealing in the furnace recrystallization occurred in the whole tested volume. What is peculiar the density of the precipitates was much higher. They were evenly distributed in grains (Fig. 10e). Around them few clusters of dislocations were observed.

In terms of the flow forming process some insights were gained during technological trials. Smaller elements (with lower diameter and height) tend to form relatively easy. The elements at various spectrum parameters did not show any tendencies to crack even at high deformation. Furthermore, their residual stress was relatively low. The mini-tensile results showed an increase of plastic flow resistance with cross section reduction and decrease in ductility. The maximum total deformation that was obtained was 67% although the value that results from the clearance between the roller and the mandrel in this case was 90%. By inducing high compressive stress like in the current case the tendency to brittle fracture was reduced. The external tensile stress must overcome the compressive residual stress before the crack tips experience sufficient tensile stress to propagate. This overall increases both the UTS and YS. It is also worth noting that the UTS for the cold deformed samples had over 1600 MPa which is a very high value even in relation with the traditional rolling process with similar strain (70%) - UTS 1510 MPa [14]. According to Woźniak [17] this was a result of lack of machine stiffness and spring back-effect. Thanks to proper planning of the second stage of the experiment a compensation effect was developed for the worked material (Inconel 625) and as well the flow forming machine (SFC 800 v500). In contrast to those results large elements proved to be significantly more challenging. High stress lead to cracking in the lower parts of the flange (Fig. 6d). This is due to overlapping of the radial forces which tend to cumulate with the height of the element. After reaching a critical value of deformation brittle cracking was seen in the formed elements. To overcome this obstacle, laser heating of the flange was used to reduce the residual stress and excess strain that was induced in the material. After heating the material for 10 min with a laser the metalworking process was resumed. There were also tests conducted with simultaneously heating and forming but they prove less effective due to heat dissipation. Additionally, linear expansion in combination with plastic deformation clamped the workpiece. This drawbacks were not described by Klocke and Brummer, [18] in their pioneering research in laser assisted metal spinning although better formability is confirmed by the current experiment.

Laser assisted metalworking is still rarely used during the forming process although the current results show that it can be successfully used for superalloys. Intermediate annealing has successfully been eliminated in the process. The material that was severely deformed regained its original good elongation and increased its YS at the cost UTS. The results are very promising in terms of future applications.

Conclusions

Thanks to the experimental methodology used in the research a large hollow cylinder, that can be used in the aerospace industry, was obtained. To achieve this laser heat-treatment was used and proper strain was selected to receive proper geometry of the final product. It is worth noting that the mechanical properties changed in a large range depending on the process parameters. The highest UTS was observed in the specimen with 70% total strain (over 1600 MPa) but the elongation was only 6% which caused cracking of the workpiece. Lower deformation caused proportionally lower increase of strength and decrease of plasticity. To change this proper heat treatments were used including and furnace annealing. The first method reduced significantly the plastic resistance (even beyond the initial state) increasing at the same time plasticity. On the other hand, furnace heating was equally effective in increasing the formability of the material although during the annealing process a large number additional precipitates were seen in the microstructure. This increased slightly the strength of the workpiece in comparison to the initial state. What is worth noting the experiment showed that by controlling the deformation and the heat treatment (especially by using the built-in laser) it is possible to acquire the required combination of strength and plasticity in cylindrical elements that can be used in the aerospace industry.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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