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Instrumented Impact Testing of Miniaturized Charpy Specimens of AM Ti-6Al-4V

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Abstract

An investigation on the impact toughness properties of wrought and additively manufactured (AM) Ti-6Al-4V was conducted at National Institute for Standards and Technology (NIST) Boulder by means of instrumented impact tests on miniaturized Charpy specimens. Full transition curves for absorbed energy and lateral expansion were obtained by performing tests in the temperature range between -196°C and 700°C . The effect of various parameters was investigated for AM specimens, namely specimen orientation, hot isostatic pressing (HIPing), and notch configuration (printed or machined). Our results indicate that AM specimens exhibit equivalent or better impact toughness than wrought material after HIPing and that the material is more resistant to cracks growing in the plane perpendicular to the build direction than in the plane containing the build direction. HIPing has a significantly beneficial effect for the AM material, while no effect of notch configuration was observed from the results obtained.

Keywords

hot isostatic pressing; impact toughness; instrumented impact tests; miniaturized Charpy specimens; Ti-6Al-4V

Introduction

Titanium alloys are known to have high mechanical resistance and toughness, even at extreme temperatures. They are lightweight and have excellent corrosion resistance and the ability to withstand extreme temperatures. However, the high cost of both raw materials and processing limit their use to military applications, aircraft, spacecraft, medical devices, highly stressed components such as connecting rods on expensive sports cars, and some premium sports equipment and consumer electronics.

Although commercially pure titanium has acceptable mechanical properties and has been used for orthopedic and dental implants, titanium in most applications is alloyed with aluminum and vanadium. These alloys have a solid solubility that varies dramatically with

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temperature, allowing it to undergo precipitation strengthening. This heat treatment process is carried out after the alloy has been worked into its final shape but before it is put to use, allowing much easier fabrication of a high-strength product.

Among titanium alloys, Ti-6Al-4V (also known as Ti 6-4) is by far the most commonly used, accounting for more than 50 % of the total titanium usage. It is an $\alpha + \beta$ alloy that is heat treatable to achieve moderate increases in strength and is recommended for use at service temperatures up to approximately 350°C. Its applications include aircraft turbine engine components, aircraft structural components, aerospace fasteners, high-performance automotive parts, marine applications, medical devices, and sports equipment. Toughness is an important consideration in the design of all these structures.

Within the investigation described in this paper, we characterized the impact toughness properties of additively manufactured (AM) Ti 6-4 by means of instrumented Charpy tests on miniaturized specimens. Tests were performed at temperatures ranging from – 196°C to 700°C in order to obtain full transition curves and calculate parameters such as ductile-to-brittle transition temperatures (DBTTs) and the upper-shelf energy (USE).

AM Charpy specimens were manufactured in two orientations (horizontal and vertical) and in two machining conditions (as-built notches and machined notches), and in hot isostatic pressed (HIPed)³ and non-HIPed state. For benchmarking purposes, these eight conditions were compared with specimens extracted from two rods of non-AM Ti 6-4 purchased from two different suppliers.

Materials and Specimens

The ten material conditions that were characterized are described in Table 1. The meaning of horizontal or vertical orientation (with respect to the build direction) is illustrated in Fig. 1.

The miniaturized Charpy specimens used for this investigation were of the reduced half-size (RHS) type, which is included in ASTM E2248-15, *Standard Test Method for Impact Testing of Miniaturized Charpy V-Notch Specimens*. Its nominal dimensions are illustrated in Fig. 2. Note that specimens with printed notches had smaller dimensions than those in Fig. 2, because their surfaces were machined to avoid excessive friction between the Charpy machine and the rough surfaces resulting from AM (three-dimensional printing). All specimen dimensions were measured before testing, including notch depth by optical profilometry (measured values were between 0.734 and 0.904 mm, with standard deviation in the order of 6 %). Values of notch root radius were not measured.

Additive manufacturing was performed using Electron Beam Melting Powder Bed Fusion equipment (Arcam A1, Mölndal, Sweden)⁴, with the following parameters: software version 3.2.132, accelerating voltage 60 kV, layer thickness 50 μm , speed factor 35. This is the

³Hot isostatic pressing is a manufacturing process by which a component is subjected to both elevated temperature and isostatic gas pressure in a high-pressure containment vessel in order to improve the material's mechanical properties and workability.

⁴Commercial names are identified in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the NIST, nor does it imply that they are necessarily the best available for the purpose.

standard build theme for Ti-6Al-4V. Gas-atomized powder with average particle diameter 70 μm (diameter range 40–100 μm) was used.

The conditions for hot isostatic pressing (HIPing) were: 2 h at 900°C, argon atmosphere, heating and cooling rates 12°C/min, pressure 100 MPa. This can be considered a standard HIPing heat treatment for Ti-6Al-4V.

The chemical composition of the characterized materials is provided in Table 2, with reference to the two non-AM conditions (B1 and B2), the AM specimens in non-HIPed condition (AMnHs), and the AM specimens in HIPed condition (AMH).

The AM specimens were split between printed and machined notches. Samples from the former group were manufactured by the AM process and subsequently milled, in order to reduce friction between specimen and machine (anvils, supports, striker) during the impact tests. However, the lateral surfaces and root of the notch were in as-manufactured (as-printed) condition and exhibited significant ruggedness and asperities, as illustrated in Fig. 3.

Conversely, specimens from the machined-notch group were manufactured by a professional machine shop from individual blanks of AM Ti-6Al-4V having dimensions of 9 by 9 by 29 mm, so that 2 mm had to be removed by milling from each surface of the original blank, followed by notching. Obviously, in this case notches had much flatter and smoother surfaces and roots.

Experimental Setup and Test Procedures

Instrumented impact tests were performed on a small-scale Charpy machine equipped with an instrumented striker. The machine has a capacity (potential energy) of 50.8 J and an impact velocity of 3.5 m/s. In accordance with ASTM E2248-15, the radius of the striking edge is 3.86 mm (nominal 4-mm striker).

Tests were performed at temperatures ranging from -196°C to 700°C . For test temperatures below room temperature (21°C), specimens were first immersed in liquid nitrogen and then transferred to the impact position in the shortest possible time. Each specimen was instrumented with a thermocouple, so that its actual temperature at the time of impact could be recorded. For tests above 21°C , the specimen temperature was monitored by a noncontact infrared temperature sensor. Up to 200°C , specimens were heated by means of a heat gun; above 200°C , a small furnace was used. In all cases, specimens were overheated by 100°C – 150°C to account for heat loss during transfer. For tests at temperatures below and above room temperature (21°C), specimens were transferred manually from the conditioning medium by means of special tongs, adapted for miniaturized specimens from the ones recommended by ASTM E23-16b, *Standard Test Methods for Notched Bar Impact Testing of Metallic Materials* for standard Charpy specimens. The transfer was initiated when the specimen temperature was way below (above) the target value, so that samples would cross the target temperature already in the impact position, and all that was required for the user was to drop the hammer by the push of a button. The overall precision in test temperature measurement can be estimated in the order of $\pm 3^{\circ}\text{C}$.

For every specimen tested, absorbed energy was measured by the encoder of the Charpy machine. Lateral expansion (i.e., increase of specimen thickness caused by plastic deformation) was measured on the broken samples by means of a caliper.

Because the specimen groups corresponding to the various conditions had slightly different cross-sectional dimensions, both absorbed energy and lateral expansion were normalized for comparison purposes according to the following procedure:

- absorbed energy (KV) was normalized by the factor Bd^2 , where B is specimen thickness and d is the ligament size, i.e., $KV_{\text{norm}} = KV/Bd^2$ [1–3];
- lateral expansion (LE) was normalized by the initial specimen thickness, i.e., $LE_{\text{norm}} = LE/B$.

Values of normalized energy (in N/mm) and lateral expansion (in percentage) were plotted and fitted as a function of test temperature by means of the commonly used hyperbolic tangent model [4]. The temperature corresponding to the inflection point of the curve is $DBTT$. The only exception was the B2 condition, for which a second-order polynomial was used for fitting on account of the limited temperature range.

From the analysis of the instrumented Charpy test records, several characteristic force values can be identified. Two of them, F_{gy} (force at general yield) and F_m (maximum force), can be analytically related for full-size Charpy specimens to the dynamic yield strength [5] and the dynamic ultimate tensile strength [6], respectively. For miniaturized Charpy specimens, no equivalent analytical or empirical relationship has been proposed yet, but it is reasonable to assume that F_{gy} and F_m remain proportional to the respective dynamic tensile properties.

Test Results

Table 3 summarizes the values of $DBTT_{KV}$ ($DBTT$ based on normalized absorbed energy), $DBTT_{LE}$ ($DBTT$ based on normalized lateral expansion), and USE_{norm} (normalized USE^5) obtained.

Several of the transition curves obtained did not show a clear plateau of normalized absorbed energy and/or normalized lateral expansion. Therefore, the corresponding values of $DBTT$ or USE_{norm} are associated with significant uncertainty and do not allow a reliable comparison between the different conditions. Hence, for comparison purposes, we decided to use primarily the values of normalized absorbed energy calculated from the transition curves at room temperature ($KV_{\text{norm}, 21^\circ\text{C}}$) and 350°C^6 ($KV_{\text{norm}, 350^\circ\text{C}}$). These values are listed in Table 3 and illustrated in Fig. 4. In the figure, bars for horizontal specimens are red and bars for vertical specimens are green. Different patterns are used for non-HIPed/HIPed specimens and for printed/machined notches.

⁵The upper shelf energy corresponds to the asymptotic plateau of the energy transition curve for high temperatures.

⁶ 350°C is generally considered the highest service temperature for Ti-6Al-4V.

Effect of Specimen Orientation

Specimens in the vertical orientation exhibited better impact toughness than those in the horizontal orientation.

The orientation effect on the Charpy transition curves for normalized absorbed energy is illustrated by the comparison of experimental data and transition curves for printed notches (Fig. 5) and machined notches (Fig. 6).

With reference to the data reported in Table 3, at room temperature (21°C) the increase in normalized absorbed energy from horizontal to vertical specimens ranges from 20–40 %, while the variation at 350°C is between –4 % (slight decrease for non-HIPed specimens with machined notches) and 21 %. The effect of specimen orientation appears less pronounced as test temperature increases.

Non-HIPed versus HIPed

HIPing tends to improve a material's mechanical properties by reducing its porosity and increasing its density [7]. For a material manufactured by additive manufacturing that is expected to exhibit significant porosity HIPing should bring significant improvements in fracture toughness.

The improvement in impact toughness is clearly visible from the results we obtained, as illustrated in terms of normalized absorbed energy for vertically manufactured specimens in Fig. 7 (printed notches) and Fig. 8 (machined notches).

In terms of normalized absorbed energy, HIPing induced an increase of impact toughness between 30 % and 76 % at room temperature and between 28 % and 68 % at 350°C.

Printed versus Machined Notches

The comparisons in terms of normalized absorbed energy between printed and machined notches in the horizontal orientation are shown in Fig. 9 (non-HIPed) and Fig. 10 (HIPed).

Although the asperities of the printed notches were expected to provide additional crack initiation sites and therefore induce lower impact toughness, particularly in the lower shelf and lower transition region, our results do not confirm these expectations. Transition curves obtained for printed and machined notches indicate substantially equivalent fracture behavior. This is confirmed by the comparison of normalized absorbed energy values in Table 3.

Nevertheless, we observe that at both 21°C and 350°C (Fig. 4), normalized absorbed energy for printed notches is higher than machined notches for all non-HIPed conditions and lower for all HIPed conditions. However, in the absence of standard deviations for test results obtained at a single temperature, we cannot comment on the statistical significance of such differences.

AM versus Non-AM (Wrought)

The results we obtained from the combined baseline data set (B1-B2, specimens extracted from two wrought bars) were compared with the different AM data sets.

Normalized absorbed energy data and transition curves are compared in Fig. 11 for non-HIPed AM conditions and in Fig. 12 for HIPed AM conditions.

Our results indicate that the impact toughness of AMnHs is similar to that of non-AM (wrought) specimens up to 200°C. In the upper transition and upper shelf regions up to 600°C, the wrought material exhibits better impact toughness properties.

After HIPing, the AM material shows better impact toughness than the wrought material (particularly in the vertical direction) up to 200°C and similar between 200°C and 600°C.

Other factors that should be considered when comparing test results between AM and non-AM specimens are the following:

1. Chemistry differences, and more specifically, oxygen and hydrogen contents (Table 2). An increase in both oxygen content (0.18 % for the baseline conditions versus 0.14 % for the AM conditions) and hydrogen content (30 ppm versus 10 ppm) is generally expected to lower the toughness of Ti-6Al-4V [8,9]. However, our results do not confirm this expected trend.
2. Microstructure differences. Baseline specimens have a mill-annealed microstructure, whereas AM specimens exhibit an acicular microstructure. This acicular (or martensitic alpha) microstructure tends to improve fracture toughness [10] and could be a contributing factor for the results obtained. More work is, however, needed to ascertain the effect of microstructure on impact toughness.

Characteristic Instrumented Forces

Our results, detailed in Ref. [11], indicate a substantial equivalence of forces at general yield and maximum forces and hence dynamic tensile properties between AM and non-AM specimens, irrespective of the notch configuration. See, for example, Fig. 13 (force at general yield) and Fig. 14 (maximum forces) for printed notches.

Conclusions

More than 100 instrumented impact tests on miniaturized Charpy specimens were performed at NIST in Boulder, CO, for the characterization of the impact toughness of Ti-6Al-4V in different conditions (wrought and additively manufactured). The effect of various parameters was also investigated (specimen orientation, HIPing, printed versus machined notches).

The main conclusions that can be drawn from this investigation are presented as follows:

- For AM specimens, we observed that the material is more resistant to cracks growing in the plane perpendicular to the build direction (vertical orientation)

than in the plane containing the build direction (horizontal orientation). Orientation effects are more significant for non-HIPed specimens and lower temperatures (below 200°C).

- HIPing reduces the porosity and increases the density of the AM material and consequently significantly improves impact toughness. The increase of normalized absorbed energy at 21°C and 350°C ranges between 30 % and 70 %, presumably as a consequence of the reduction in available fracture initiation sites.
- The configuration of the Charpy specimen notch (printed or machined) does not have a significant effect on the impact toughness of AM specimens, despite the ruggedness and asperities observed in printed notches.
- Below 200°C, the impact toughness of AM specimens is similar to that of wrought material in the non-HIPed condition, while the wrought material performs better at higher temperatures. After HIPing, AM specimens exhibit better impact toughness than wrought material below 200°C, and the properties appear similar between 200° C and 600°C.
- Based on instrumented forces at general yield and maximum forces measured from instrumented impact tests, the dynamic tensile properties of AM and wrought specimens appear similar, irrespective of notch configuration, specimen orientation, and post-manufacturing heat treatment (HIPed or non-HIPed).

References

1. Lucon, E, McCowan, CN, Santoyo, RL. NIST Technical Note 1858. U.S. Department of Commerce; Washington, DC: 2015. Impact Characterization of 4340 and T200 Steels by Means of Standard, Sub-Size and Miniaturized Charpy Specimens.
2. Lucon, E, McCowan, CN, Santoyo, RL. NIST Technical Note 1865. U.S. Department of Commerce; Washington, DC: 2015. Impact Characterization of Line Pipe Steels by Means of Standard, Sub-Size and Miniaturized Charpy Specimens.
3. Lucon E, McCowan CN, Santoyo RL. Overview of NIST Activities on SubSize and Miniaturized Charpy Specimens: Correlations with Full-Size Specimens and Verification Specimens for Small-Scale Pendulum Machines. J Pressure Vessel Technol. 138(3)2016; :031405.
4. Oldfield W. Fitting Curves to Toughness Data. J Test Eval. 7(6)1979; :326–333. DOI: 10.1520/JTE11508J
5. Server WL. General Yielding of Charpy V-Notch and Precracked Charpy Specimens. J Eng Mater Technol. 100(2)1978; :183–188. DOI: 10.1115/1.3443469
6. Lucon E. Estimating Dynamic Ultimate Tensile Strength from Instrumented Charpy Data. Mater Des. 972016; :437–443. DOI: 10.1016/j.matdes.2016.02.116
7. Atkinson HV, Davies S. Fundamental Aspects of Hot Isostatic Pressing: An Overview. Metall Mater Trans A. 31(12)2000; :2981–3000. DOI: 10.1007/s11661-000-0078-2
8. Ferguson, RR, Berryman, RG. Report AFML-TR-76-137. Rockwell International; Los Angeles: 1976. Fracture Mechanics Evaluation of B-1 Materials.
9. Henry, SD, Davidson, GM, Lampman, SR, Reidenbach, F, Boring, RL, Scott, WW, Jr, editors. Fatigue Data Book: Light Structural Alloys. ASM International; Materials Park, OH: 1995. Titanium Alloys Fatigue and Fracture; 183–204.
10. Froes, FH, editor. Titanium—Physical Metallurgy, Processing, and Applications. ASM International; 2015. Mechanical Properties and Testing of Titanium Alloys; 113–140.

11. Lucon, E, Hrabe, N. NIST Technical Note 1936. U.S. Department of Commerce; Washington, DC: 2016. Instrumented Impact Tests on Miniaturized Charpy Specimens of Additively Manufactured (AM) Ti6Al4V.

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FIG. 1.
Illustration of building directions for miniaturized AM Charpy specimens.

FIG. 2.
Nominal dimensions for RHS Charpy specimens.

FIG. 3. Stereomicrograph of the printed notch of a miniaturized Charpy specimen (vertical direction).

FIG. 4.

Normalized absorbed energies at 21°C (a) and 350°C (b) for the investigated conditions. MH = machined notch, horizontal direction; MV = machined notch, vertical direction; PH = printed notch, horizontal direction; PV = printed notch, vertical direction; (XX)H = HIPed; (XX)nH = non-HIPed.

FIG. 5.
Normalized absorbed energy data and transition curves for specimens with printed notches.

FIG. 6. Normalized absorbed energy data and transition curves for specimens with machined notches.

FIG. 7.
Comparison between non-HIPed and HIPed specimens (printed notches, vertical direction).

FIG. 8.
Comparison between non-HIPed and HIPed specimens (machined notches, vertical direction).

FIG. 9.
Comparison between printed and machined notches for non-HIPed, horizontal specimens.

FIG. 10.
Comparison between printed and machined notches for HIPed, horizontal specimens.

FIG. 11.
Normalized absorbed energy for AM non-HIPed conditions and non-AM conditions.

FIG. 12.
Normalized absorbed energy for AM HIPed conditions and non-AM conditions.

FIG. 13.
Forces at general yield for baseline and AM (printed notches) specimens.

FIG. 14.
Maximum forces for baseline and AM (printed notches) specimens.

TABLE 1

Summary of the different Ti 6-4 conditions investigated.

Condition	AM?	HIPed?	Notches	Orientation	Notes
B1	No	No	Machined	Long axis: parallel to rod axis,	Purchased from supplier 1
B2	No	No	Machined	Notch: radial	Purchased from supplier 2
PHnH	Yes	No	Printed	Horizontal (see Fig. 1)	–
PVnH	Yes	No		Vertical (see Fig. 1)	–
PHH	Yes	Yes		Horizontal (see Fig. 1)	–
PVH	Yes	Yes		Vertical (see Fig. 1)	–
MHnH	Yes	No	Machined	Horizontal (see Fig. 1)	–
MVnH	Yes	No		Vertical (see Fig. 1)	–
MHH	Yes	Yes		Horizontal (see Fig. 1)	–
MVH	Yes	Yes		Vertical (see Fig. 1)	–

TABLE 2

Chemical composition of the characterized materials (weight %).

Condition	Al	C	Fe	H	N	O	V	Ti
B1	6.58	0.02	0.250	0.003	0.010	0.180	4.3	Bal.
B2	6.39	0.03	0.165	0.003	0.025	0.175	4.0	Bal.
AMnH	5.89	0.01	0.160	0.001	0.020	0.140	4.4	Bal.
AMH	5.82	0.01	0.170	0.001	0.020	0.140	4.3	Bal.

TABLE 3

Summary of results obtained from instrumented Charpy tests.

Condition	Orientation	HIPed?	$DBTT_{KV}, ^\circ C$	$DBTT_{LE}, ^\circ C$	$USE_{norm}, N/mm$	$KV_{norm, 2J^*C}, N/mm$	$KV_{norm, 350^*C}, N/mm$
Baseline [B1-B2]	–	No	220.7	200.8	288.9	57.6	245.4
PHnH	Horizontal	No	187.7	202.8	173.1	57.7	155.1
PVnH	Vertical		102.6	141.4	208.6	76.1	175.9
PHH	Horizontal	Yes	209.0	218.4	247.0	78.1	197.8
PVH	Vertical		189.2	139.0	252.6	99.3	238.5
MHnH	Horizontal	No	197.4	328.6	194.8	56.2	151.4
MVnH	Vertical		119.5	147.0	184.5	67.3	144.9
MHH	Horizontal	Yes	215.0	92.0	305.4	84.7	201.7
MVH	Vertical		46.6	89.1	264.4	118.5	243.5