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Experimental validation of miniaturized specimen developed to perform uniaxial tensile test on high performance materials

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Abstract. This work deals with the experimental validation of the proposed specimens geometries which design process through Finite Element Method was presented during AIAS 2019 and published afterwards [1]. In particular, non-conventional miniaturized specimen geometry compliant to ASTM E8 and ISO 6892, specifically developed to work with MaCh3D [2], an innovative miniaturized tensile testing machine, were considered. The size-reduction of specimens is advantageous both in terms of material and equipment: smaller specimens require lower forces to be broken, hence a compact tensile testing machine can perform the mechanical characterization activity. In this experimental validation, mechanical properties determined using the three proposed MaCh3D miniaturized specimens are compared to full-size ISO 6892 samples. Specimens were cut from AISI 304 stainless steel plates, with different thickness according to specimens requirements. Good accordance has been found between the standard and non-standard geometries.

1. Introduction

The development of a miniaturized specimen, compatible with MaCh3D testing system, is a viable method to assess high performance materials mechanical properties. Such a methodology is very interesting especially for metal Additive Manufacturing (AM), where the cost of raw material is high and therefore there is great interest in limiting the quantity needed for its characterization [3–6].

From literature reviews reported in the works of Gu et al [7], Frazier [8] and Lewandowski and Sefi [9] amongst others, it has been found that materials such as Ti6Al4V or Inconel 718 can easily reach and exceed a tensile strength of 1000 MPa. It must be noted that the same raw material, adopted in different technologies, such as DMLS and EBM, for example, presents different mechanical properties. Hence, cross section area of the full-size MaCh3D specimen, measuring 39 mm^2, must be reduced to one equal (or lower) 5 mm^2 to exceed tensile strength of such high performing alloys, given the maximum exercisable load of 5 kN.

The dimensions of tensile test samples are labeled in many standards as *proportional* or *non-proportional*, as reported in the work of Loveday [10]. In the former, the gauge length (L_0) is set in proportion to the square root of the cross-sectional area (S_0), which may be either rectangular as well as cylindrical. Gauge length is thus defined as $L_0 = k_s\sqrt{S_0}$ where k_s is a suitable constant (very often 5.65) for sheet specimen and $L_0 = k_{ro}D_0$ for round specimen, being k_{ro} the same constant for circular

sections and D_0 the original section diameter. The 5.65 constant value derives from the expression $5\sqrt{4/\pi}$, which will provide the same cross-sectional area as a cylindrical specimen where the gauge length is set at five times the diameter.

The proportional requirement is based on the principle that geometrically similar specimens should deform similarly, although the formulation allows a departure from strict similarity in terms of width/thickness ratio etc.

1.1. Specimen geometry

Specimen geometry have to be compatible with MaCh3D fixtures. In particular, reduced section length had to be connected to specimen droplet-shaped heads, the latter being unmodified in respect to full size MaCh3D specimen.

The final specimen geometry, which development through Finite Element Method (FEM) has been reported in the work of Bergonzi et al. [1], defined as "S-Size" is reported in Figure 1 (a) with main dimensions: L_c is the reduced section length, W represent specimen width, T specimen thickness. The maximum obtainable stress level on the reduced section, given the maximum applied load of 5 kN, is equal to 1587.30 MPa, that is enough to exceed tensile strength for most AM and traditional materials. G-2 spline fillets were adopted, since the stress concentration at the beginning of the straight section is reduced in comparison to round ones. Following the same procedure of miniaturization, two more geometries were developed, having larger cross sections and defined as "M-Size" and "L-Size", intended to be used with less performing material or in the case a small section such as the one of S-Size specimen cannot be produced. Main dimensions are reported in Figure 1 (b) and (c), respectively. Some more practical rather than analytical limits were imposed upon initial gauge length and specimen thickness. The former, in order to have enough space to be able to mount a clip-gage extensometer, the latter limiting thickness to standard values for metal sheets. In fact, S-Size, M-Size and L-Size specimens are developed mainly for AM technologies but can be used also to assess mechanical properties of metal foils. So, in order to not over complicate specimen production, thickness of 1.5 mm, 2.0 mm and 3.0 mm were established for the three geometries, allowing to cut samples directly from the bulk sheet.
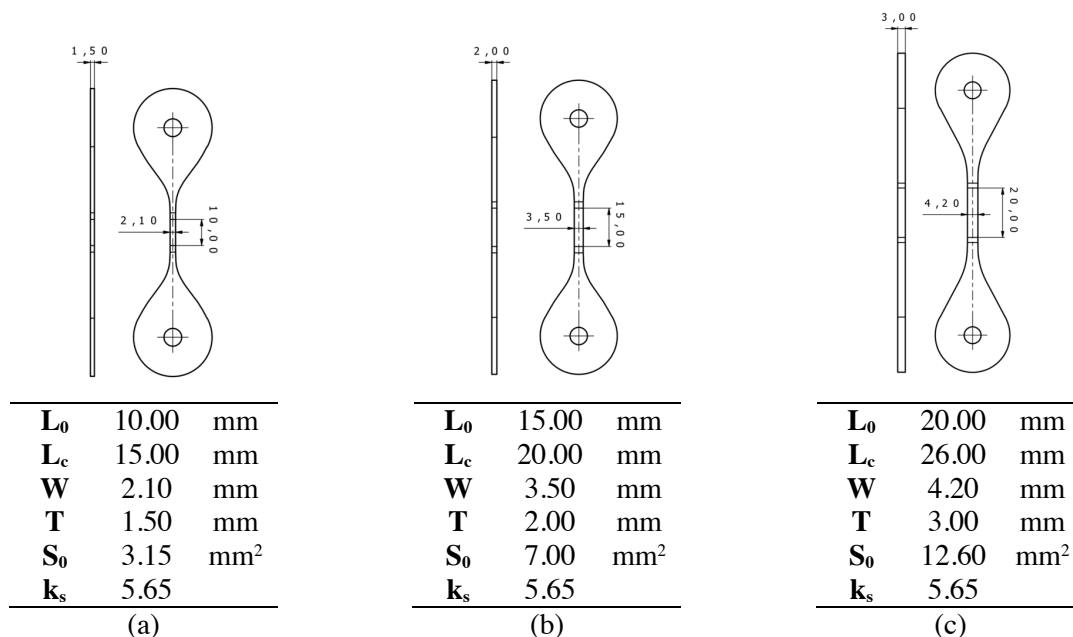
	(a)			(b)			(c)	
L_0	10.00	mm	L_0	15.00	mm	L_0	20.00	mm
L_c	15.00	mm	L_c	20.00	mm	L_c	26.00	mm
W	2.10	mm	W	3.50	mm	W	4.20	mm
T	1.50	mm	T	2.00	mm	T	3.00	mm
S_0	3.15	mm^2	S_0	7.00	mm^2	S_0	12.60	mm^2
k_s	5.65		k_s	5.65		k_s	5.65	

Figure 1. MaCh3D miniaturized specimens: S-Size (a), M-Size (b), L-Size (c).

2. Experimental campaign

To validate numerical results, an experimental campaign was conducted using AISI 304 stainless steel specimens cut from cold rolled sheet.

Before to invest in an additive material characterization campaign, due to its high costs, this preliminary activity allowed to obtain reliable results at a fraction of the cost and time occurred to produce samples. Moreover, AISI 304 is a widely used material and can be compared to different sources across literature in order to double-check results. In addition, this would be a typical usage scenario for MaCh3D as a tool to asses constant production quality of metal sheets.

2.1. Materials and methods

Specimens were produced starting from three different AISI 304 stainless steel sheet thicknesses in order to represent the three developed geometries, which thickness are 1.5, 2.0 and 3.0 mm for S-Size, L-Size and M-Size respectively. The different amount of cold work for different thicknesses due to metal rolling, is known to determine different mechanical properties in the material [11–13] and in particular an increase in mechanical tensile strength and decrease in ductility. For this reason, for each sheet thickness, ISO 6892 [14] standard samples, were produced: validation has been carried out between ISO and MaCh3D geometries for a given sheet thickness; the specimens used are depicted in Figure 2.
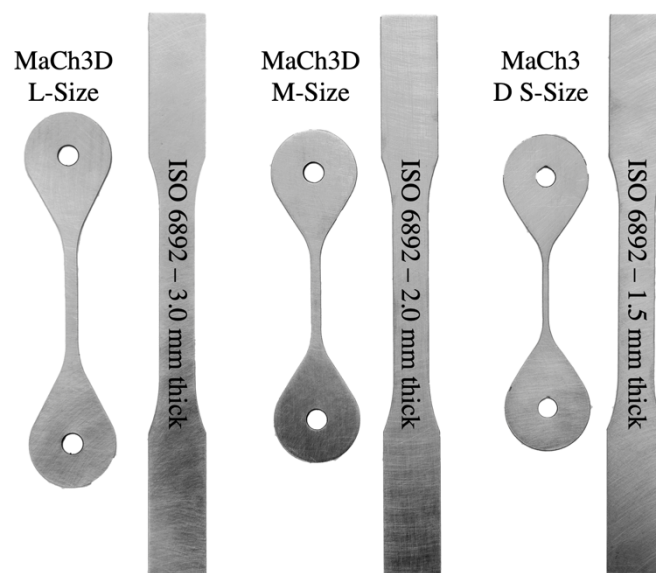

Figure 2. ISO and MaCh3D specimens used for experimental campaign.

Crosshead speed was determined according to ISO 6892 Method A2 which involves the control of the estimated strain rate over the parallel length for open loop systems (e.g. strain rate cannot be controlled using an extensometer due to testing system limitation). In this case, estimated strain rate over the parallel length ($\dot{\varepsilon}_{LC}$) was $0.002 \ s^{-1}$ for all considered geometries.

AISI 304 is a quite tough material with elongation at failure up to 40%. During this experimental campaign, the extensometer gauge length (L_e) was kept as long as possible (50mm in the case of ISO samples) in comparison to specimen straight section length, as specified from ISO 6892. However, the available extensometer, MTS 632.12F-24 (MTS Systems Corporation, Eden Prairie, USA) allows to determine strains up to 8.5% on 50 mm gauge length because of travel limitation. The same issue is present also in the case of MaCh3D specimens that, even though having shorter gauge lengths, did not allow to acquire the entire stress-strain curve. For this reason, comparison curves between ISO and MaCh3D specimen are plotted up to 8.5% only: a qualitative analysis is offered comparing strain values

obtained from crosshead displacement. In Table 1, different specimen gauge lengths and extensometer base lengths are reported together with maximum detectable strain (ε_{max}).

Table 1. Extensometer base length and maximum detectable strain according to specimen geometry.

Spec. geometry	$\mathbf{L_0}$ [mm]	$\mathbf{L_e}$ [mm]	$\boldsymbol{\varepsilon_{max}}$ [%]
ISO	50	50	8.5%
MaCh3D S-Size	10	10	40%
MaCh3D M-Size	15	15	27%
MaCh3D L-Size	20	20	20%

The limited strain measurement allowed, nonetheless, to determine elastic modulus as well as 0.2% plastic extension proof strength. Five repetitions were performed for each geometry, resulting in 30 valid tests; an MTS 810 (MTS Systems Corporation, Eden Prairie, USA) servo-hydraulic machine with 100 kN loadcell was used together with special adapters to accommodate MaCh3D proprietary pulling heads, as depicted in Figure 3 (a) and (b): the extensometer was carefully aligned with specimen loading axis and its own weight was partially balanced by hanging the connection cable to the machine frame.
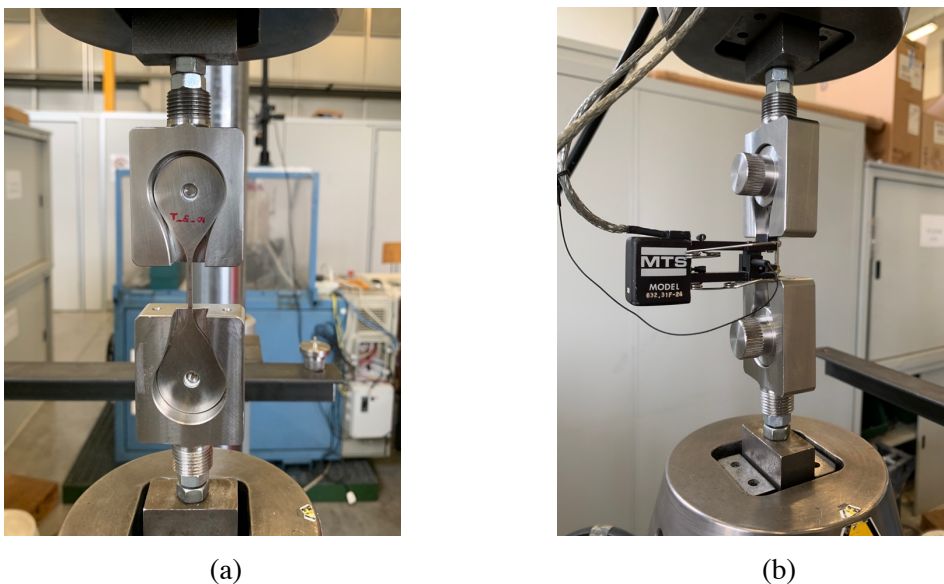

(a)   (b)

Figure 3. S-Size specimen housed into MaCh3D pulling heads (a) and with caps and extensometer mounted (b).

The motivation to use a "traditional" tensile testing machine instead of MaCh3D lies in the fact that in this way all the samples can be tested on a single frame, thus eliminating machine architecture influence on the test results, as it was done during full-size specimen validation [2]. Due to the different specimens thickness, adapters disks, depicted in Figure 4, were used in order to maintain specimen middle plane in line with the loadcell: in fact, default sample seat in MaCh3D pulling heads has a calibrated depth to host 3 mm thick specimens. In the case of 1.5 mm specimens, two 0.75 mm depth disks were used, whilst for the 2 mm specimens, disks of 0.5 mm were adopted.
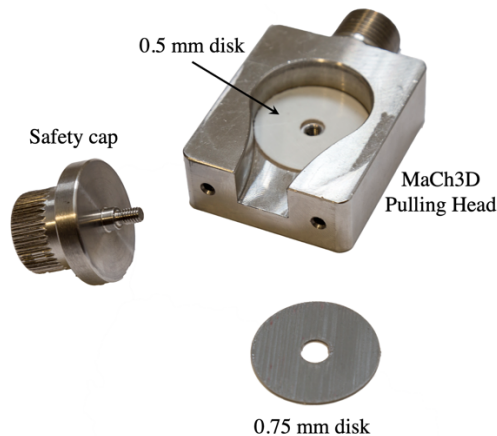
Figure 4. Shimming disks and MaCh3D pulling head.

3. Results

Stress-strain curves comparison between ISO standard specimens and MaCh3D are reported in Figure 5 (a), (b) and (c), all showing very good agreement between the two sets of data, while in Table 2 average results together with standard deviation (in brackets) are reported.
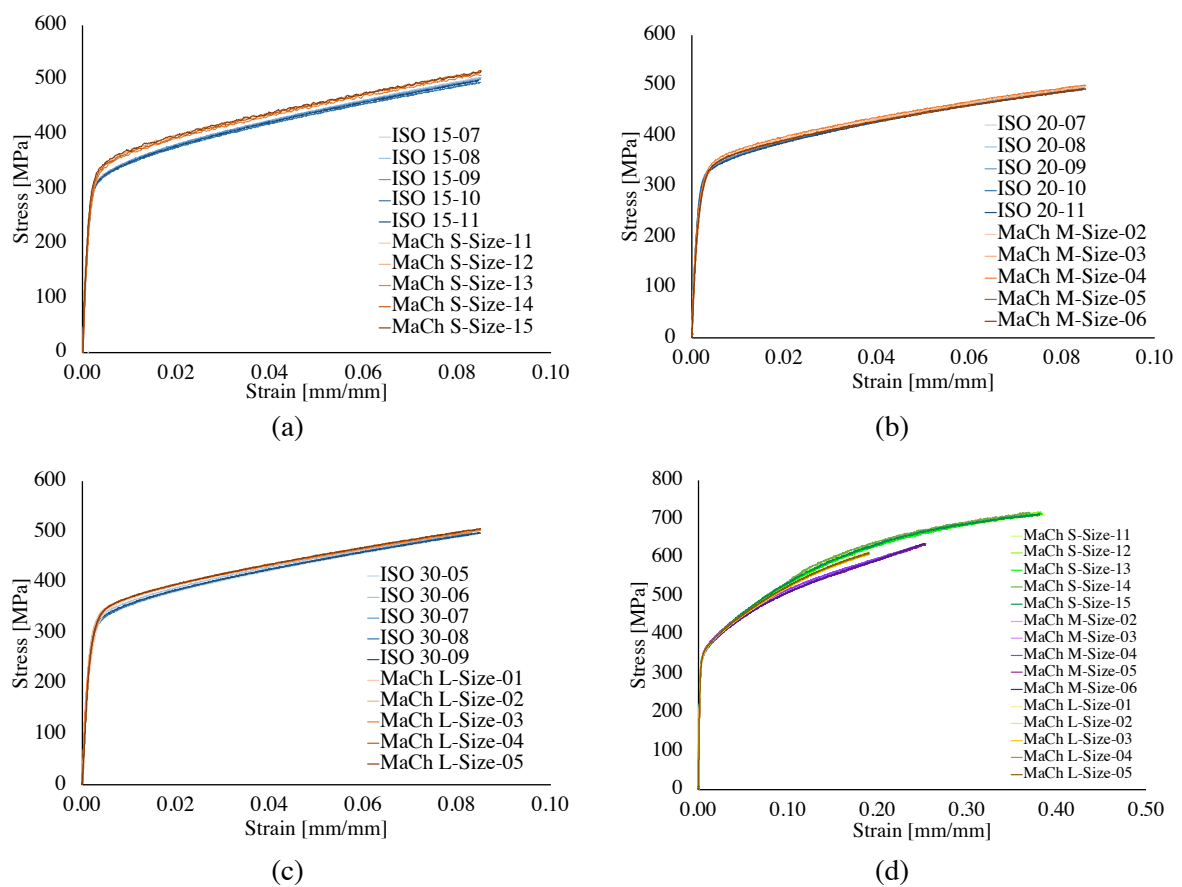

Figure 5. Stress strain curves comparison for ISO and MaCh3D specimens: S-Size (a), M-Size (b), L-Size (c). Comparison between all MaCh3D curves up to ε_{max}(d).

In all cases, MaCh3D specimens presents higher R_m values respect to reference ISO samples. This could be due to a small size effect, still present despite section proportional scaling. Moreover, it must be noted that MaCh3D specimen are free to move inside pulling heads, allowing very good alignment of the reduced section in respect to load train, whereas ISO specimen are clamped into machine hydraulic grips and even if they were carefully positioned, slight misalignment can be still present producing early specimen failure. Maximum difference in terms of tensile strength between the two standards geometries is 3.7% noticed for the M-Size samples: a value well in line with other comparative studies between full size and miniature specimens [15,16] and in any case within the expected experimental scatter.

Regarding tensile strength, the three sheets present different values, according to ISO 6892. The same behavior is detected using MaCh3D specimens: this aspect is fundamental since it demonstrated the ability of the proprietary sample geometry to identify a different material behavior.

Elastic modulus maintains its value throughout all sheet thickness, confirming the trend reported in the work of Milad et al. [11] where the effect of cold rolling on AISI 304 sheet is studied. To accurately evaluate its value, the rectilinear trait of the stress-strain curve has been isolated and least squares linear regression has been performed until a value of the correlation coefficient (r^2) of 0.995 or better was found.

Table 2. ISO vs MaCh3D specimens results. 1.5 mm (a), 2.0 mm (b), 3.0 mm thickness (c).

	E	R_m	$R_{p\,0.2}$		E	R_m	$R_{p\,0.2}$		E	R_m	$R_{p\,0.2}$
	[MPa]	[MPa]	[MPa]		[MPa]	[MPa]	[MPa]		[MPa]	[MPa]	[MPa]
ISO 6892 1.5 mm	200561 (1054)	715.25 (2.03)	316.95 (2.27)	**ISO 6892** 2.0 mm	200376 (308)	741.06 (1.53)	330.80 (1.45)	**ISO 6892** 3.0 mm	200445 (2649)	691.01 (2.03)	324.48 (4.37)
MaCh3D S-Size	203014 (4052)	728.12 (5.69)	332.74 (4.27)	**MaCh3D** M-Size	201775 (707)	769.53 (2.64)	330.73 (4.05)	**MaCh3D** L-Size	199984 (2676)	713.17 (1.25)	331.05 (3.59)
Δ %	1.2%	1.8%	4.7%	**Δ %**	0.7%	3.7%	0.0%	**Δ %**	-0.2%	3.1%	2.0%
	(a)				(b)				(c)		

Proof strength at 0.2% plastic deformation does not present a clear trend, both for ISO specimens and MaCh3D, the latter showing almost constant values throughout all specimens with low variation (the highest being 4.7% in the case of S-Size specimen) in respect to the ISO ones.

Huge difference is found in terms of the maximum force reached, thanks to specimen reduced section. In particular, S-Size specimen is the only geometry that virtually could have been used to test the material on MaCh3D tensile testing machine, given the maximum load capacity of 5 kN, presenting an average maximum tensile force of 2180.51 N. The M-Size, designed to reach 714 MPa on the middle section at machine full capacity, fall short outside the allowable load.

Even if the extensometer strain is not acquired throughout the stress-strain curve, a qualitative idea of maximum elongation can be grasped considering crosshead displacement.

Calculating the strain from crosshead displacement, considering the distance between the two grips as base length, in the case of ISO specimens being equal to 95mm, values of 41.8%, 43.0% and 40.3% are obtained for 1.5, 2.0, 3.0 thicknesses, respectively. The calculated value is affected by errors due to machine compliance, deformation of specimens inside grips, non-perfect positioning etc. but gives an overall idea of the maximum uniform elongation. In the case of MaCh3D specimens, to evaluate strain starting from crosshead displacement is not straightforward since the clearance between specimen heads and seats affects results. However, as reported Figure 5 (d), the maximum average strain registered using extensometer in the case of MaCh3D S-Size specimens reaches values of 39.8%, well in line with material data sheet and values determined in the case of ISO samples.

Comparing ISO and MaCh3D specimens up to the same level of strain, as in Figure 6, a slightly higher dispersion is noticeable in the latter specimen geometry.
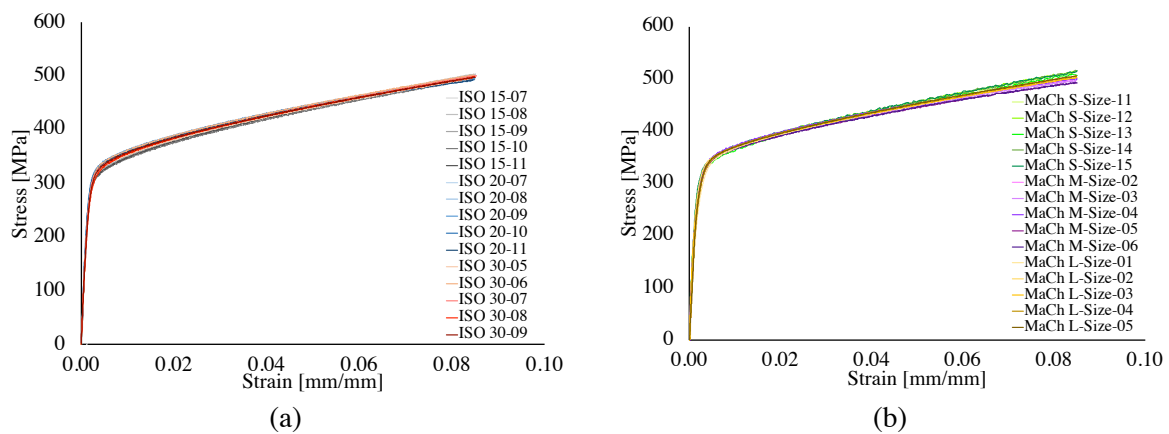
Figure 6. ISO (a) and MaCh3D (b) Stress-Strain curves compared up to the same strain value.

Fractured surfaces have been photographed using a digital microscope (Dino-lite, Almere, Netherlands) with 30x magnification and reported in Figure 7. As it can be seen, in all case, ductile fracture takes place, showing marked necking deformation at rupture.
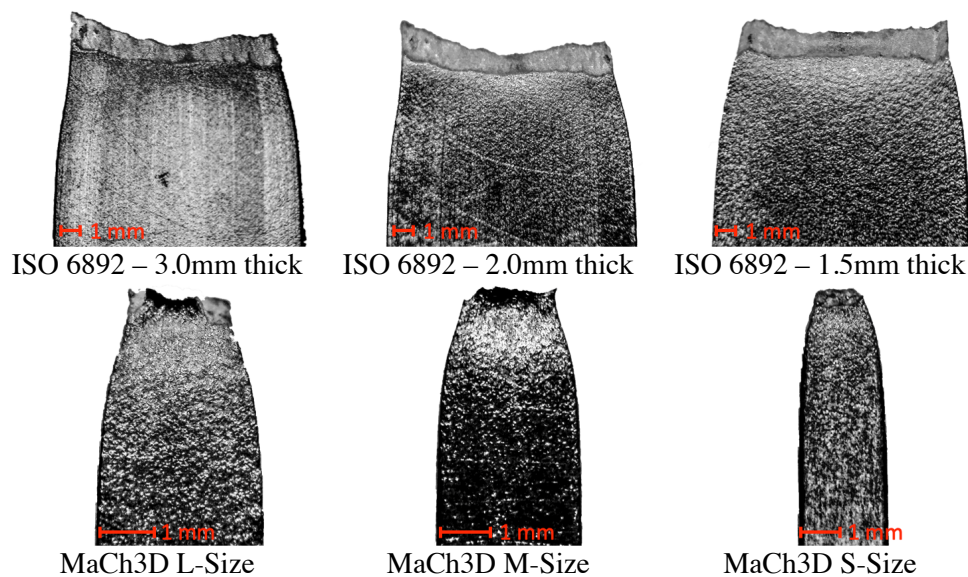

Figure 7. Fracture zone for different specimens.

4. Conclusions

Three different miniaturized specimen geometries have been developed and numerically validated in terms of stress distribution in the parallel section and at the end of the spline fillet.

A G2 cubic interpolation spline has been used as transition curve, allowing to decrease stress concentration factor at the end of the fillet, without altering specimen working principle based on droplet heads interference with the custom designed ma-chine fixture. A preliminary experimental campaign conducted using AISI 304 stainless steel confirmed the numerical results, by assessing compatibility of the miniaturized specimens with the ones obtained according to ISO 6892. Further characterization activities need to be implemented to assess complete specimen compatibility with metal AM fabrication methods.

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