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Qualifying Bulk Metallic Glass Gear Materials for Spacecraft Applications

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Abstract

NASA is evaluating bulk metallic glass (BMG) gears for extreme environment (cryogenic) applications; e.g., Europa Lander. The main purpose of this report is to recommend a quality assurance (QA) protocol for the production of consistent and reliable gear castings. Currently, there are two, separate manufacturers involved; Materion Corporation produces re-melt stock and Visser Precision Cast, Inc. produces BMG castings. Division of the existing alloy specifications document into material supplier-specific documents is proposed. Until a composition-based specification is established, processability- and performance-based methodologies may be an option during manufacturing development. It is recommended that each of the documents has a distinct focus; the processing behavior of crystalline feedstock, and the mechanical behavior of amorphous castings.

The report addresses the “gray area” in QA testing between these materials suppliers by outlining an equitable division of responsibilities. The use of a number of dedicated reference dies to produce witness materials for physical and mechanical property evaluation is strongly recommended. Allocation of QA tests between screen testing of gear castings, and formal testing of witness materials, including cryogenic properties, is also proposed. The most effective suite of tests for both physical and mechanical property determination are down-selected from available national and international standards. The contents of this report create a solid basis for selection of QA tests, thereby setting the stage for negotiations between the materials suppliers and the customer.

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Symbols and Abbreviations

Symbols

at.%	percentage by atom
d_{max}	critical casting thickness
ppm	parts per million
T_g	glass transition temperature
T_l	liquidus temperature
T_x	crystallization temperature
ΔT_x	differential between T_x and T_g
τ_x	onset of crystallization time
wt.%	percentage by weight

Abbreviations

ASTM	American Society for Testing and Materials
CT	compact tension
DMA	dynamic mechanical analysis
DSC	differential scanning calorimetry
DTA	differential thermal analysis
FTR	fracture toughness ratio
GFA	glass forming ability
IIT	instrumented indentation testing
ISO	International Standards Organization
JPL	Jet Propulsion Laboratory
KSC	Kennedy Space Center
LaRC	Langley Research Center
NDE	non-destructive evaluation
PG	planetary gear
QA	quality assurance
SENB	single edge notch bend
SWG	strain wave gear
TMA	thermomechanical analysis

TTT ....time-temperature-transformation

XRD ....X-ray diffraction

1. Introduction

1.1. Background

Deployment of planetary gears (PGs) and strain wave gears (SWGs) on spacecraft began with the Apollo 15 Lunar rover and continues with the Mars Curiosity rover, figure 1(a). Historically, most of the SWGs, also known as harmonic drives, used by NASA on spacecraft are lubricated steel [ref. 1]. Select future missions, such as to the Europa moon orbiting Jupiter, will operate at very low temperatures, figure 1(b). Such environments will necessitate heating of incumbent lubricants for equipment functionality. This creates a strong impetus to eliminate wet lubrication in order to drastically reduce vehicular power consumption and battery weight requirements. The Jet Propulsion Laboratory (JPL) is pioneering the use of bulk metallic glass (BMG) gears as a way to eliminate the need for wet lubrication of gearboxes on spacecraft [ref. 2]. Representative SWG and PG assemblies, along with the constituent components are shown in figure 2(a) and 2(b), respectively. Prototype castings of BMG gears are presented in figure 3, which reveals the small dimensions of the components. It should be noted that the cup-shaped, flexspline gear shown has a varying section thickness of ≤ 2 mm.

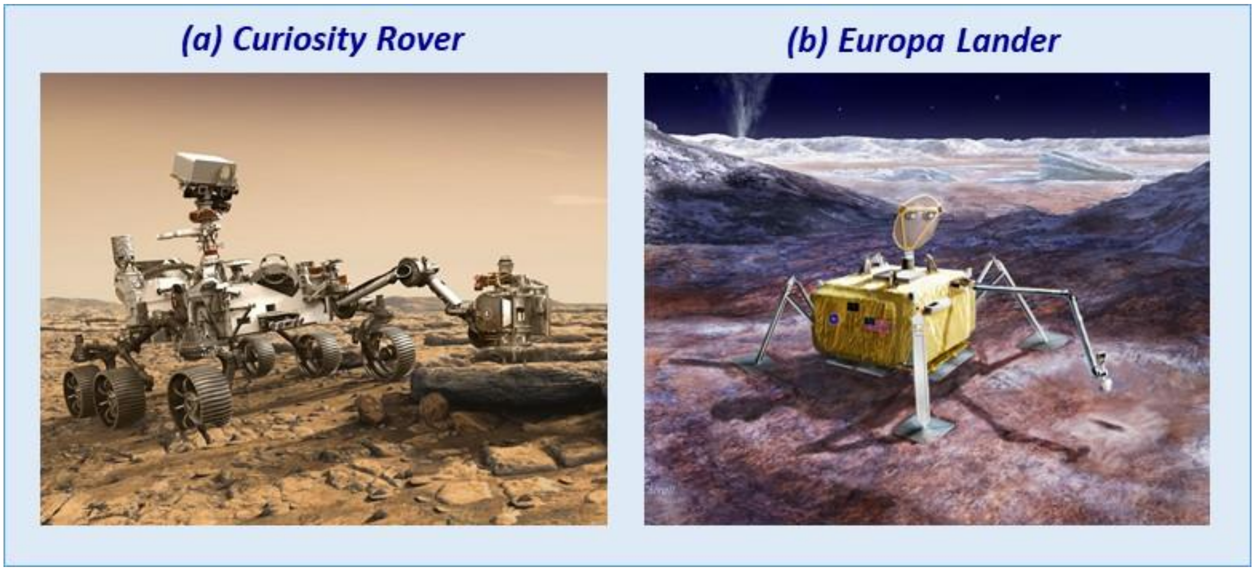


Figure 1. Existing and planned NASA spacecraft that operate with strain wave and planetary gears: (a), Curiosity rover on Mars (current); (b), Europa lander on a Jupiter moon (future).

BMGs are not widely-used in engineering applications and structural component manufacture is an emerging technology [ref. 3]. The critical properties for gear materials are high wear and fatigue resistance, which may be correlated with high hardness and toughness. A common mode of failure in gears is cracking at the roots of teeth [ref. 4]. Quantifying fracture toughness provides a measure of resistance to such crack initiation and propagation. A material property that is unique to the flexspline component of SWGs is high resilience (yield strength^2/elastic modulus). BMGs are ideal candidates for this application because they exhibit high hardness, high strength and low modulus [ref. 5]. The Achilles heel may be fracture toughness, which varies widely with composition and temperature. However, high toughness at ambient temperature has recently been achieved in copper/zirconium-based BMGs by severely limiting the oxygen content [ref. 6]. The wear resistance under ambient conditions is also 60% better than the steel gears currently used on Curiosity rover.

Figure 2. Representative assemblies and components for gearboxes on NASA spacecraft that can employ precision castings: (a), strain wave gears; (b), planetary gears.

This report addresses some of the issues associated with the acceptance of BMGs as structural materials in harsh environments. The Langley Research Center (LaRC) is serving as an independent technical authority to advise JPL during manufacturing development. The Kennedy Space Center (KSC) is responsible for certification of gear assemblies, including vacuum and lubricant effects. On this project, JPL and LaRC are tasked with qualification of materials by quantifying physical and mechanical properties. JPL and KSC are tasked with certification of individual components by conducting simulated-service testing. The goal of the combined effort is to advance the technology readiness level for application on a variety of future missions. Throughout, the report refers to the Agency as the “customer” and tends not to differentiate between the various NASA facilities involved.

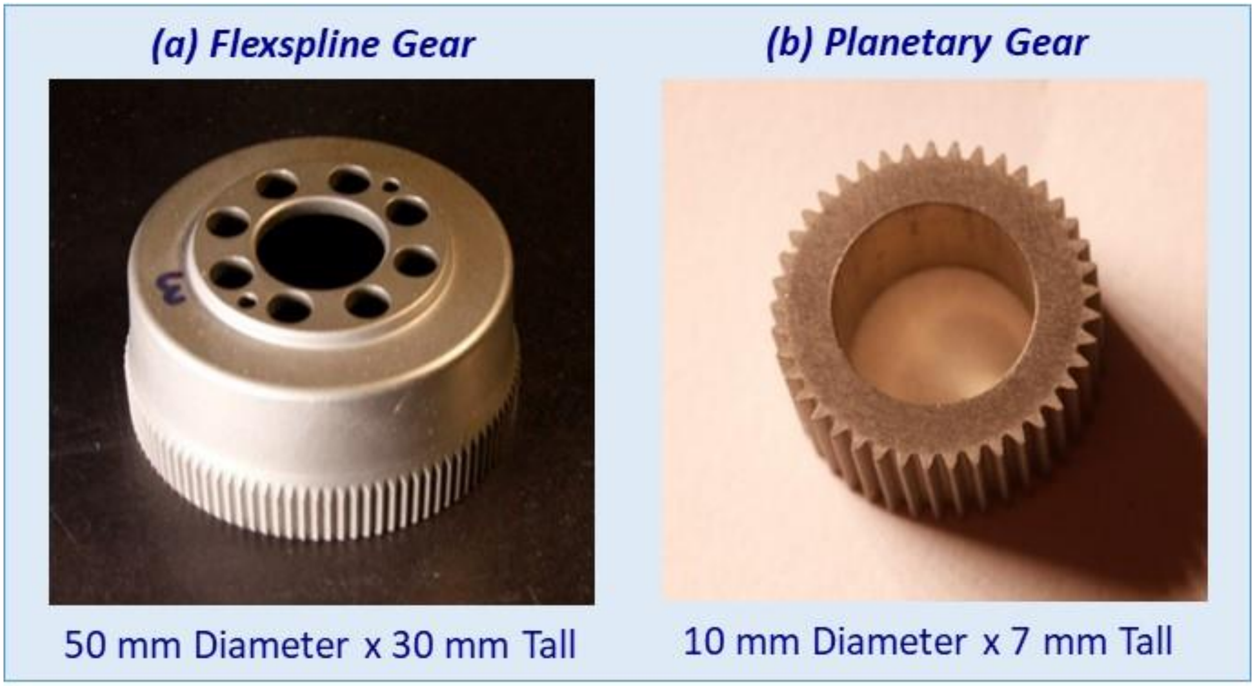


Figure 3. Prototype castings of BMG gear components: (a), flexspline gear; (b), planetary gear.

1.2. Manufacture of BMG Gears

Currently, manufacturing of BMG gears is a dual-step process with responsibilities divided between separate materials suppliers, as shown in figure 4. The first step involves combining pure metals in the correct proportions to create a master alloy in the form of crystalline ingots. The process involves vacuum induction melting and casting into chilled copper molds as 19 mm diameter rods. ***Materion, Beryllium & Composites*** in Elmore, OH**,** is the current manufacturer of the rod stock, figure 4(a) [ref. 7]. These ingots become re-melt feedstock for the subsequent amorphous casting process. The fabrication of BMG gears may best be described as a hybrid of die casting (of metals) and injection molding (of polymers). Referred to as “amorphous alloy injection molding casting,” ***Visser Precision Cast*** in Denver, CO**,** is the current manufacturer of SWGs, figure 4(b) [ref. 8]. The target compositions and some typical properties for the candidate BMG alloys are assembled in table 1 [refs. 1, 2, 9, 10, and 11]. Composition is critical as alloy content governs both the ability to cast fully-amorphous material and the mechanical behavior of the final product. Key intrinsic material properties for future gear applications comprise hardness, modulus, yield strength and toughness [ref. 12]. S77PT ($Zr_{43}Cu_{43}Al_7Be_7$, at.%) is the current alloy of choice for manufacturing the flexspline component in SWGs [ref. 2].

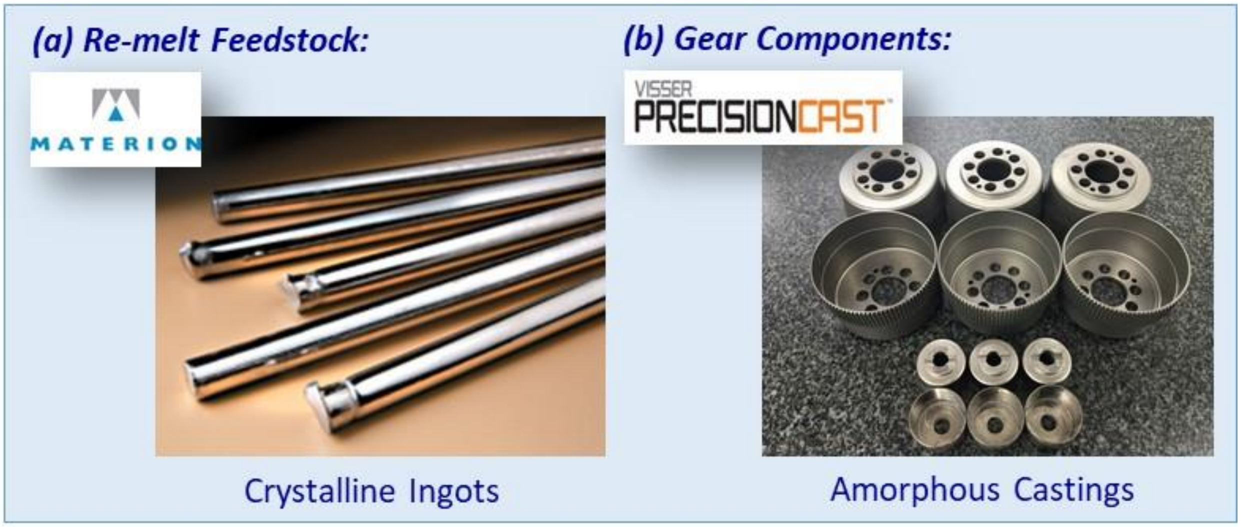
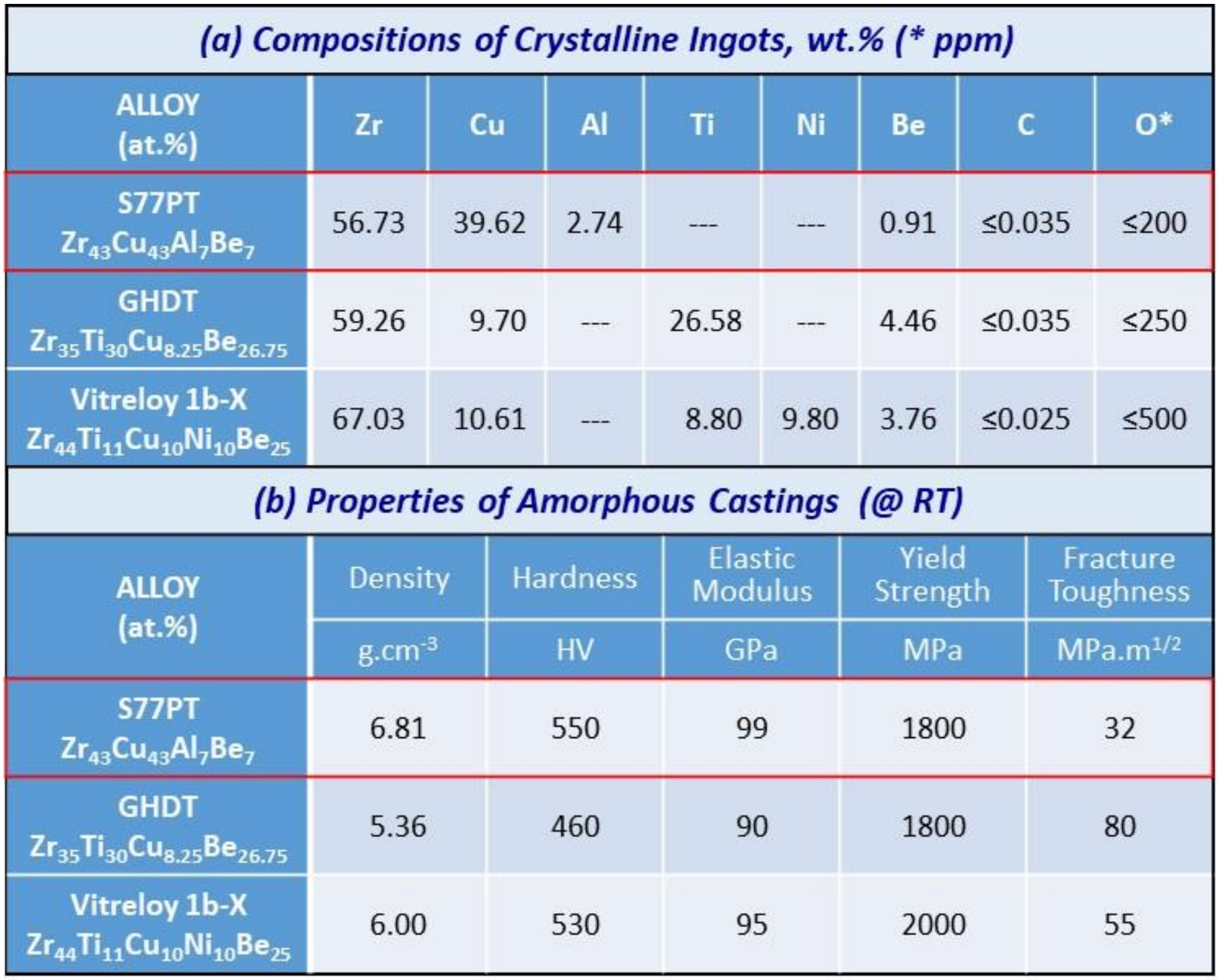


Figure 4. BMG gear manufacture involves two materials suppliers: (a), re-melt feedstock, in the form of crystalline ingots; (b), gear components, in the form of amorphous castings.

1.3. Production of BMG Castings

There are three intrinsic material properties that play a vital role in part quality when the crystalline feedstock is re-melted to produce BMG castings. These comprise the glass forming ability (GFA), the temperature-dependent viscosity, and onset of crystallization (incubation) time of the molten alloy [ref. 13]. A “time-temperature-transformation” (TTT) diagram is a quantitative way to envision the thermal tolerances required to produce BMGs during solidification processing. Figure 5 shows a TTT diagram representative of zirconium-based BMGs exhibiting the classic C-shaped curve [ref. 14]. The liquidus temperature, T_l, defines the lower limit of the equilibrium liquid region. The crystallization temperature, T_x, defines the lower limit at which crystals can nucleate. The glass transition temperature, T_g , defines the upper limit for the amorphous solid region. The C-shaped curve, defines the boundary for the crystalline solid region. This region must be completely avoided in order to produce amorphous materials.

Table 1. Target compositions and properties for candidate BMG alloys: (a), crystalline ingot compositions; (b), amorphous casting properties [refs. 1, 2, 9, 10, and 11].



(a) Compositions of Crystalline Ingots, wt.% (ppm)*

ALLOY (at.%)	Zr	Cu	Al	Ti	Ni	Be	C	O*
S77PT $Zr_{43}Cu_{43}Al_7Be_7$	56.73	39.62	2.74	---	---	0.91	≤0.035	≤200
GHDT $Zr_{35}Ti_{30}Cu_{8.25}Be_{26.75}$	59.26	9.70	---	26.58	---	4.46	≤0.035	≤250
Vitreloy 1b-X $Zr_{44}Ti_{11}Cu_{10}Ni_{10}Be_{25}$	67.03	10.61	---	8.80	9.80	3.76	≤0.025	≤500

(b) Properties of Amorphous Castings (@ RT)

ALLOY (at.%)	Density	Hardness	Elastic Modulus	Yield Strength	Fracture Toughness
	$g.cm^{-3}$	HV	GPa	MPa	$MPa.m^{1/2}$
S77PT $Zr_{43}Cu_{43}Al_7Be_7$	6.81	550	99	1800	32
GHDT $Zr_{35}Ti_{30}Cu_{8.25}Be_{26.75}$	5.36	460	90	1800	80
Vitreloy 1b-X $Zr_{44}Ti_{11}Cu_{10}Ni_{10}Be_{25}$	6.00	530	95	2000	55

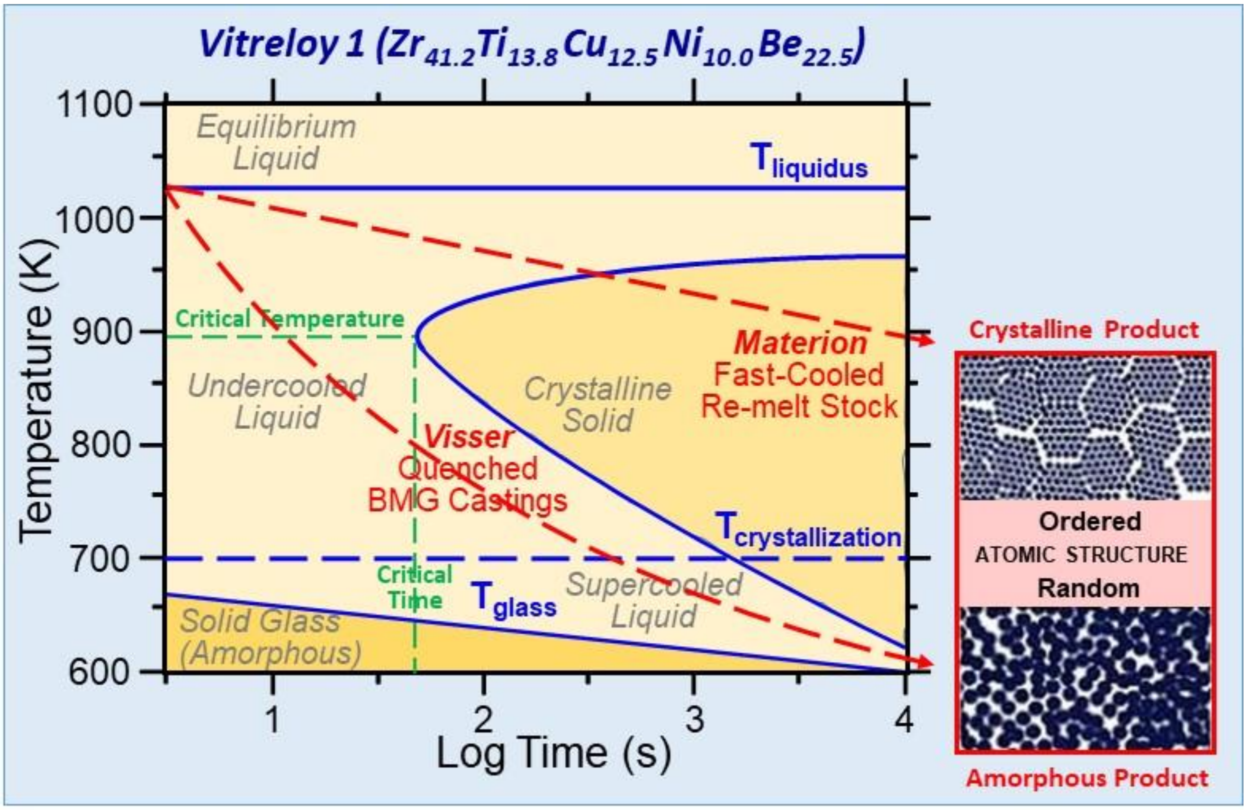


Figure 5. TTT diagram representative of zirconium-based BMG alloys [ref. 14]. Location of the "C" curve nose is critical during solidification processing. Difference in cooling profile between production of crystalline ingots and amorphous castings is highlighted.

Figure 5 also illustrates the difference in casting practices between the crystalline material produced by ***Materion*** and the amorphous material produced by ***Visser***. The "nose" of the C-curve defines the maximum time permitted to reach the nose temperature without nucleating crystals in the molten metal. Knowing the location of the nose is critical because it defines the minimum cooling rate during the casting of BMG gears. Crystalline product exhibits an ordered atomic structure. In contrast, amorphous product exhibits a random atomic structure. This means that BMGs do not contain many of the features that govern mechanical behavior in conventional alloys; e.g., dislocations and grain boundaries. The biggest benefit of this characteristic is that amorphous materials can be 3-4 times stronger than their crystalline counterparts.

The fluidity of the molten metal as the temperature decreases is also important for conformance of the casting with the die geometry [ref. 15]. Viscosity, the inverse of fluidity, increases as the liquid transitions from the "equilibrium," through the "undercooled," to the "supercooled" zone [ref. 14]. The differential between the crystallization temperature and the glass transition temperature, ΔT_x, is also critical. It defines the upper and lower bounds of the supercooled liquid region and governs the thermal tolerances for viscous material flow during cooling.

Consequently, the diagram depicts the process control required to produce fully-amorphous, fully-formed castings. A high cooling rate avoids the crystallization "nose" in the early stage, and adequate fluidity ensures complete die filling in the latter stage.

1.4. Materials Specifications Datasheets

The existing materials specifications datasheet is JPL D-56223, Rev. A; "*Detail Requirements for S77PT Bulk Metallic Glass, a Copper, Zirconium, Aluminum, Beryllium Alloy*". In this "draft" document, specifications for composition, physical and mechanical properties are interspersed between crystalline and amorphous product. Consequently, it is recommended that the current document be replaced by two "preliminary" documents that distinguish between re-melt feedstock and gear castings. In targeting the individual manufacturers, it is important that both documents have the same chemical composition specifications. The trace element content, which exerts a strong influence on both GFA and fracture toughness is critical [ref. 2]. Beyond elemental composition, the ***Materion*** document needs to concentrate on processing characteristics of the feedstock, and the ***Visser*** document needs to focus on intrinsic material properties of the BMG gears.

1.5. Primary Specification – Composition

Monitoring the alloy composition at all stages of BMG gear manufacturing is paramount; i.e., crystalline ingots and amorphous castings. The solute content of fugitive elements, such as aluminum, and trace elements, such as oxygen, are particularly affected by solidification processing. Consequently, measuring and reporting the composition is the responsibility of both ***Materion*** and ***Visser*** for all materials supplied. Samples should be randomly extracted from batches of ingots and castings with chemical analyses conducted per guidelines selected from the following American Society for Testing and Materials (ASTM) standards:

- ASTM B824; Standard Specification for General Requirements for Copper Alloy Castings [ref. 16]
- ASTM E88; Standard Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition [ref. 17]
- ASTM E478; Standard Test Methods for Chemical Analysis of Copper Alloys [ref. 17]

Alloy composition may ultimately become the lone specification, but "composition-processing-performance" correlations must first be established. Currently, the target content for the primary elements is known for alloy S77PT, but the margins have yet to be defined for each element. Acceptable ranges for zirconium, copper, aluminum, and beryllium need to be determined. This will require the compilation of a database that contains the effects of all compositional variances on both processability and performance. Maximum allowables for trace element content should also be established. It is known that an oxygen content of < 500 ppm is required, and values closer to 200 ppm are desirable, for adequate GFA and fracture toughness [ref. 6].

2. Qualification Strategy

2.1. Strategy Adopted for Qualifying BMG Gear Materials

This report culminates from a thorough assessment of quality assurance (QA) testing as it relates to BMG gear manufacture. The evaluation of intrinsic material properties under monotonic loading conditions is a prerequisite. Cyclic or simulated-service testing, including fatigue properties, wear behavior and lubricant effects, are not covered. After establishing compositional variances, each of the two materials specification documents needs to have a distinct focus:

- Processability of crystalline ingots from ***Materion***:
  The emphasis should be on the physical properties relevant to the castability of the re-melt feedstock. The use of reference dies to produce "acceptance" castings that provide witness materials for determining GFA, viscosity and inclusion content is proposed.
- Performance of amorphous castings from ***Visser***:
  The emphasis should be on the mechanical properties relevant to the function of the BMG gears. The use of reference dies to produce "qualification" castings that provide witness materials for determining stiffness, strength, and toughness is proposed.

The most effective approach calls for both screen testing and formal testing. Formal tests involve complex specimen preparation and specialized test equipment, so the prime responsibility resides with the ***Customer***. The data generated are suitable for establishing "composition-processing-property" correlations. In contrast, screen tests involve readily-available and established methods, such that responsibility can reside with the ***Materials Suppliers***. The philosophy is one of discriminatory testing; i.e., "pass/fail," based on specifications derived from formal testing.

A key decision entails an equitable division of testing responsibilities between the ***Materials Suppliers*** and the ***Customer***. Whether categorized as screen or formal tests, QA testing should adhere to standardized procedures as much as possible. This may include drawing on both ASTM and International Standards Organization (ISO) guidelines developed for unrelated materials, such as ceramics, composites or glasses. From a practical viewpoint, most QA data will be generated under room temperature conditions. However, establishing the relationship between ambient and cryogenic temperature properties should be a requirement given the projected service conditions.

2.2. Acceptance Castings and Qualification Castings

The production of witness materials is common practice in the die casting industry [ref. 18]. It is suggested that all witness materials be cast in dedicated reference dies. It is imperative that the designs duplicate the processing characteristics of the die used to fabricate gear components. Two categories of tooling are proposed in order to produce

- Acceptance castings for evaluation of processing characteristics; e.g., GFA, viscosity; and
- Qualification castings for determination of mechanical properties; e.g., tensile, toughness.

The term "acceptance" castings is selected to imply assessment of the suitability of crystalline feedstock to produce fully-amorphous, fully-formed castings. The term "qualification" castings refers to evaluation of the physical and mechanical properties of amorphous castings. In both instances, the case for employing dedicated reference dies for QA purposes is illustrated in figure 6 [ref. 19]. Flexspline gears for SWGs comprise small, complex-shaped, thin-walled castings, figure 6(a). Extracting flat specimens with adequate dimensions is problematic for QA testing.

Die cavity designs, molten metal flow, solidification and cooling rates need to duplicate those optimized for casting gears. Therefore, casting witness materials in a "combination die" is not considered a viable option, figure 6(b). The added volume of appendages, modified runners and gating patterns will affect these variables. Material characteristics may be compromised and not be truly representative of the fabricated parts. As a consequence, employing a dedicated "multiple-cavity die" is recommended as the best option, figure 6(c).

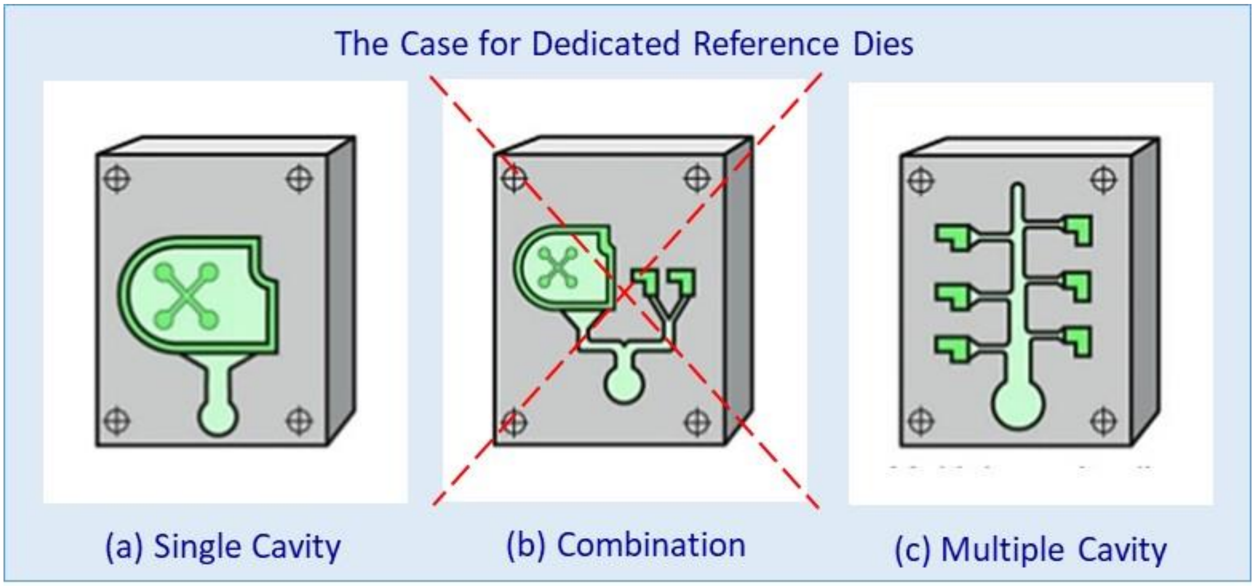


Figure 6. Adaptation of tooling configurations routinely employed in the die casting industry [ref. 19]: (a), single cavity – to produce parts only; (b), combination – to produce parts and witness materials simultaneously; (c), multiple cavity – to produce witness materials only.

It is imperative that the production of witness materials for QA testing employs casting practices that simulate BMG gear manufacturing. The heat transfer characteristics of the die and the casting parameters exert a strong influence on solidification behavior [refs. 20 and 21]. The melt volume and section thicknesses of acceptance and qualification castings should be comparable to fabricated components. It is suggested that QA tests for processability should be conducted prior to the mass production of gear components. Similarly, QA tests for performance should be conducted prior to certification of gear assemblies. It is recommended that responsibility for producing all witness materials resides with ***Visser***. This approach is logical for two reasons; (i), casting parameters, such as molten metal cooling and flow rates, are proprietary, and (ii), die designs, such as runner and gate geometries, are competition-sensitive.

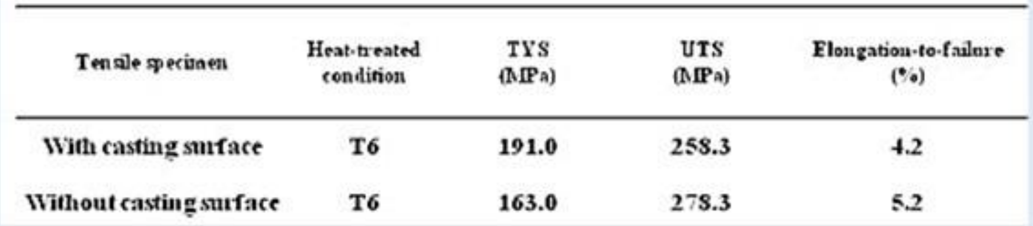
2.2.1. Casting of Mechanical Test Specimens

Although the casting of amorphous metals is more akin to injection molding, it is still germane to adopt practices from the casting of crystalline metals. An example is presented in figure 7(a) for permanent mold casting of multiple tensile test specimens [ref. 22]. A group of round test bars is cast close to final dimensions and then each is machined to ASTM specifications. Even though crystalline materials tend to be less sensitive to surface flaws, the test data in figure 7(b) indicate the differences that can be encountered as a result of surface machining. The ultimate strength and ductility increase, but the yield strength decreases for an aluminum alloy in the T6 temper condition. As a consequence, this effect should be a major consideration during specimen preparation of BMG materials for mechanical testing.

An issue unique to BMGs is that they can behave as much like a glass as they can a metal. Such ceramic-like materials tend to be notch-sensitive, requiring an extremely smooth surface finish on mechanical test specimens. Traditional machining methods used for sizing of specimens can compromise test results. Milling, EDM, or water jet operations can introduce surface flaws that cause premature failure, both inside and outside of the prescribed gage section. Extensive hand polishing of test sections is frequently required for accurate data. An elegant solution comprises taking advantage of the near-net-shape capability of the current casting methodology. The injection molding approach is renowned for reproducing fine details and adhering to tight tolerances. Figure 8(a) illustrates that multi-cavity dies are routinely employed in the amorphous castings industry [ref. 23]. Figure 8(b) reveals that multiple tensile blanks can readily be produced via die casting [ref. 24]. Therefore, the opportunity exists to produce groups of BMG test specimens so close to ASTM specifications that subsequent surface preparation is minimized.

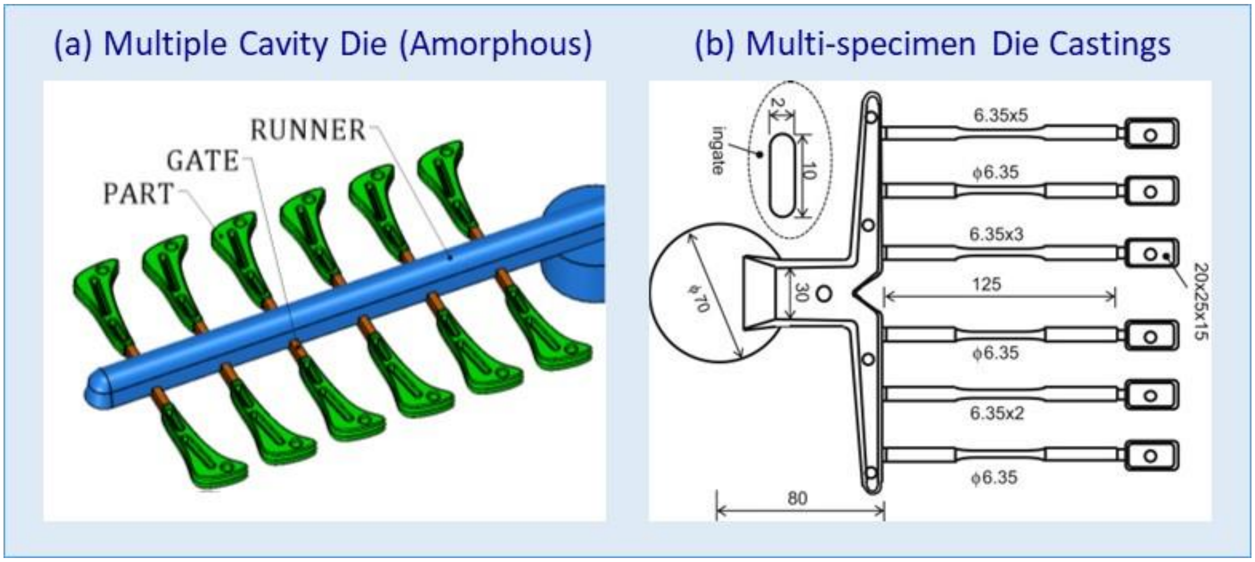
(a) Multi-specimen Castings (Crystalline)

(b) Specimen Preparation Effects on Properties



Tensile specimen	Heat-treated condition	TYS (MPa)	UTS (MPa)	Elongation-to-failure (%)
With casting surface	T6	191.0	258.3	4.2
Without casting surface	T6	163.0	278.3	5.2

Figure 7. Influence of surface machining on mechanical testing [ref. 22]: (a), group of round test bars cast close to final dimensions; (b), difference in tensile properties between as-cast and machined test specimens.



(a) Multiple Cavity Die (Amorphous)

(b) Multi-specimen Die Castings

Figure 8. Opportunity to cast groups of BMG test specimens close to ASTM dimensions: (a), multi-cavity dies already used in the amorphous castings industry [ref. 23]; (b), multiples of tensile specimens already produced by die casting [ref. 24].

2.2.2. Casting of Notched Test Specimens

There is also an opportunity to exploit the fine detail reproducibility of the casting process to fabricate notched mechanical test specimens. This would facilitate fracture toughness testing by reducing post-fabrication machining requirements, reducing surface flaws, and improving data accuracy. Figure 9(a) shows the configuration for Izod and Charpy impact specimens customarily used during screen testing [ref. 25]. Figure 9(b) illustrates the choices of Charpy notch geometry, the selection depending on projected fracture resistance [ref. 26]. The same philosophy applies to formal testing that includes single edge notch bend (SENB) and compact tension (CT) test specimens. Figure 10(a) shows the typical specimen configurations for toughness testing in tension and bending [ref. 27]. Figure 10(b) indicates that the notch geometry is more complex and is designed to allow for fatigue pre-cracking and to accommodate clip gages [ref. 28]. Even reducing the amount of machining required would be an obvious benefit during specimen preparation.

(a) Impact Testing Specimens

(b) Charpy Notch Geometries

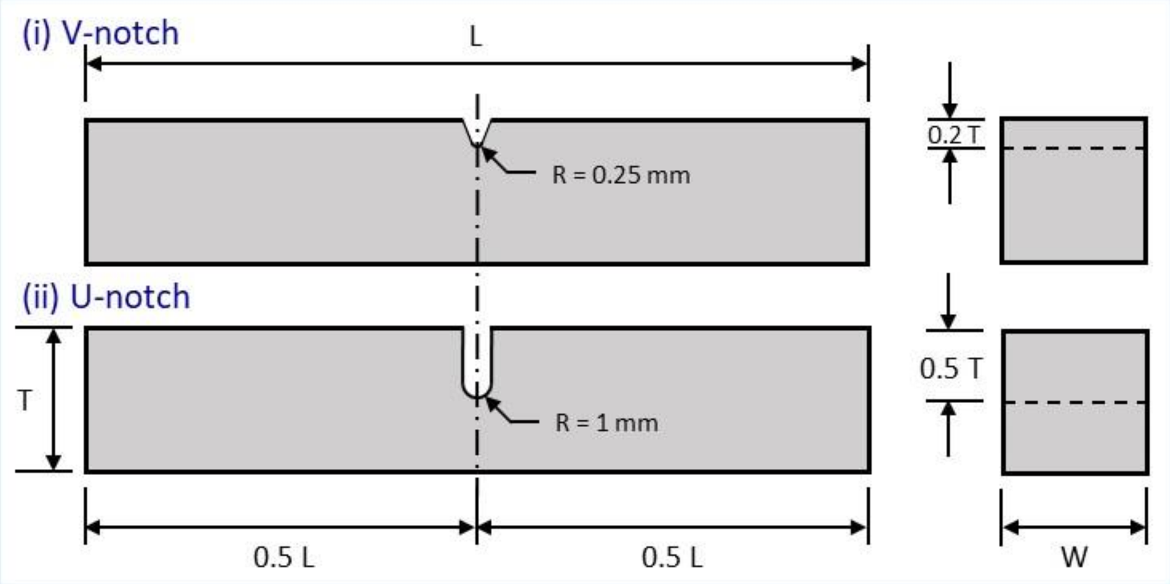
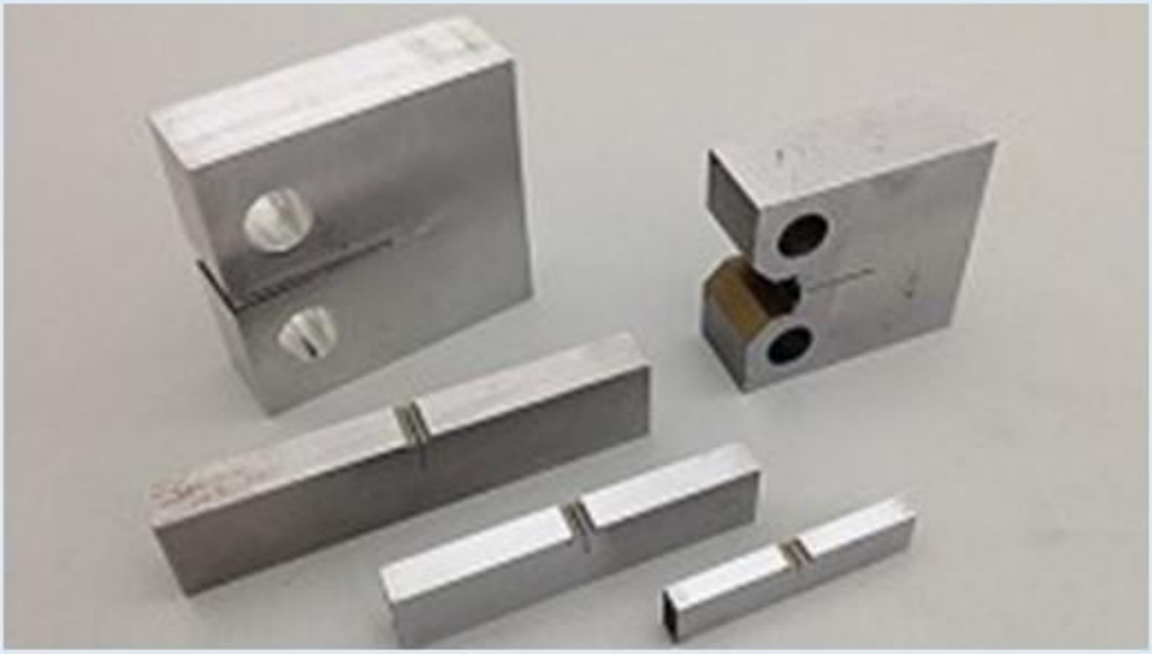


Figure 9. Opportunity to cast notched screen testing specimens close to ASTM dimensions: (a), impact test specimen configurations - (i), Izod, and (ii), Charpy [ref. 25]; (b), common choices of notch geometry for Charpy testing [ref. 26].

(a) Compact Tension & Single Edge Notch Bend Testing



(b) Notch Geometries

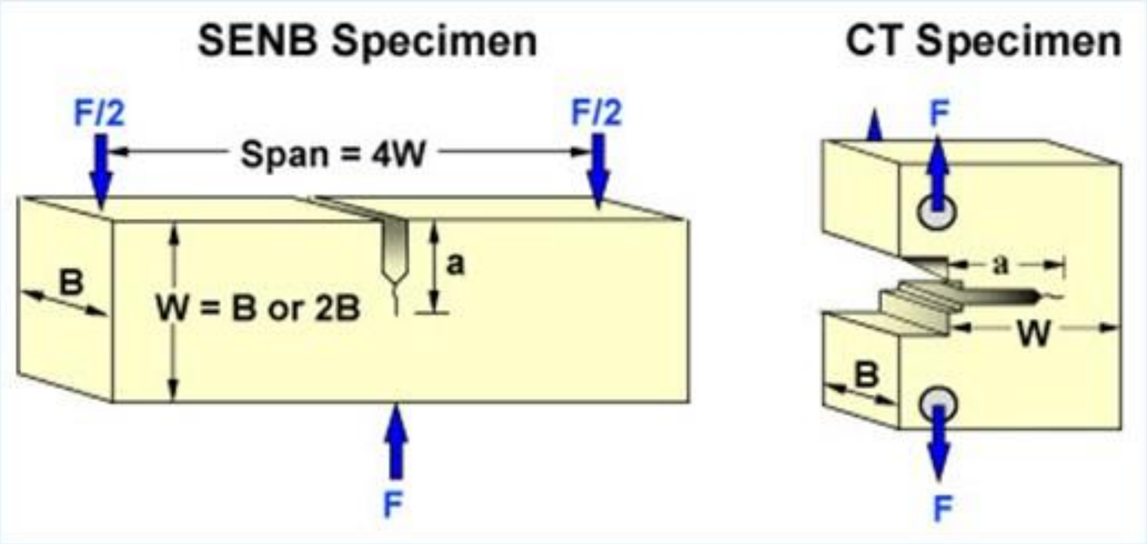


Figure 10. Opportunity to cast notched formal testing specimens close to ASTM dimensions: (a), compact tension (CT) and single edge notch bend (SENB) specimen configurations [ref. 27]; (b), common notch geometries for SENB and CT testing [ref. 28].

2.3. Cryogenic Testing of BMG Gear Materials

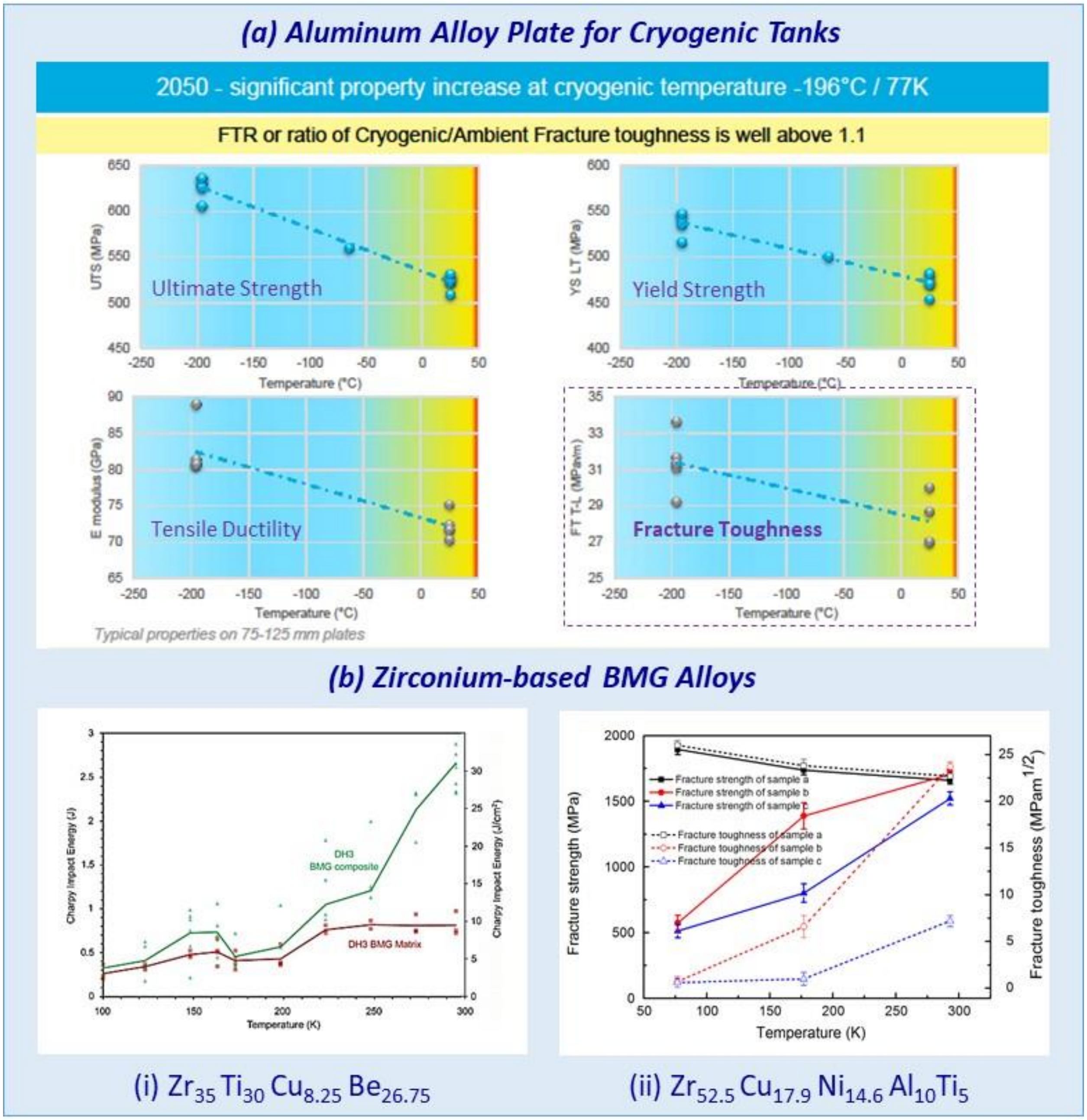
Current specifications regarding the environmental conditions for QA testing are realistic from the perspective of ***Materials Suppliers***. JPL D-56223, Rev. A states that "*Unless otherwise indicated, all tests shall be performed at 75°F ± 5°F (24°C ± 3°C)*". However, BMG gears are being developed for cryogenic applications on future missions. For example, the projected target for Europa Lander is operation at $T \leq 100K$ (-170°C) for 10 hours. Obviously, evaluating the cryogenic properties of BMG gear materials should be "otherwise indicated," but such testing should be confined to the realm of ***Customer*** responsibility.

A parallel situation involves qualification of materials for manufacturing of cryogenic fuel tanks on launch vehicles. Fabricated tanks may be certified at ambient temperature only after a positive fracture toughness ratio (FTR) has been demonstrated for the materials at cryogenic temperatures [ref. 29]. For example, Figure 11(a) shows recent data for aluminum-lithium alloy 2050 plate manufactured by Constellium in Ravenswood, WV [ref. 30]. Along with ultimate/yield strength and ductility, fracture toughness values increase from room temperature to liquid nitrogen temperature. Ultimately, this mechanical behavior provides the necessary property margins that are required for manned flight certification.

Cryogenic property data for zirconium-based BMG alloys are sparse in the literature and data conforming with ASTM E399 are not encouraging [e.g., ref. 31]. Pertinent data are shown in figure 11(b) and 11(c) for $Zr_{35}Ti_{30}Cu_{8.25}Be_{26.75}$ and $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_{5}$ (at.%) alloys [refs. 32 and 33]. Figure 11(b) contains Charpy impact energy data as a function of temperature in the 100-300K range for a BMG composite compared with the matrix alone. Ambient temperature is 298K and liquid nitrogen temperature is 77K. Attention is drawn to the red line (DH3 BMG matrix) that refers to monolithic matrix material. The data show that fracture energy decreases from 9.1 $J.cm^{-2}$ at room temperature to 2.9 $J.cm^{-2}$ at liquid nitrogen temperature. These results imply a negative FTR in the cryogenic temperature regime.

Figure 11(c) shows the trend in measured fracture strength and calculated fracture toughness at 75K, 175K, and 300K. Attention is drawn to the black line (sample a) that refers to material in the as-cast condition. The data reveal that the fracture toughness increases from ~ 22.5 $MPa.m^{1/2}$ at room temperature to ~ 26.0 $MPa.m^{1/2}$ at liquid nitrogen temperature. These results indicate a positive FTR in the cryogenic temperature regime. It is important to note that the oxygen content is not reported in either case, and tramp elements exert a strong influence on fracture behavior [refs. 2 and 6].

These results highlight the importance of creating a cryogenic property database for alloy S77PT for extreme environment service. The data compiled can be correlated with ambient temperature properties in order to validate QA tests conducted at room temperature. In the absence of cryogenic toughness data, evidence is currently lacking that zirconium-based BMG alloys have the damage tolerance required to perform in the targeted applications.



(a) Aluminum Alloy Plate for Cryogenic Tanks

2050 - significant property increase at cryogenic temperature -196°C / 77K

FTR or ratio of Cryogenic/Ambient Fracture toughness is well above 1.1

Typical properties on 75-125 mm plates

(b) Zirconium-based BMG Alloys

(i) $Zr_{35}Ti_{30}Cu_{8.25}Be_{26.75}$

(ii) $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$

Figure 11. Establishing the fracture toughness ratio (FTR) between cryogenic and ambient temperatures: (a), aluminum alloy 2050 for cryogenic tank applications [ref. 30]; (b), zirconium-based BMG alloys – (i), JPL/DH3 [ref. 32], and (ii), Vitreloy 105 [ref. 33].

3. Processability Testing

3.1. The "Gray Area" in the Materials Supply Chain

Visser is ultimately responsible for establishing the acceptance/rejection criteria for the crystalline feedstock provided by ***Materion***. By necessity, the protocol during manufacturing development is different than that adopted for a mature materials supply chain. Although the target compositions of BMG alloys are known, acceptable composition variations have not been defined. This scenario dictates the manufacture of prototype batches of amorphous castings prior to mass production of BMG gears. Consequently, the responsibilities for processability testing are divided between the two ***Materials Suppliers***. The situation represents the "gray area" in the current materials supply chain that is subject to debate. The final product needs to meet the performance specifications provided by the ***Customer***, but success hinges on first meeting the processability specifications.

3.2. Acceptance Castings for Processability Evaluation

The role of acceptance castings is dedicated to verification of the processing characteristics of the crystalline ingots produced by ***Materion*** during the manufacturing development phase. The nominal composition of alloy S77PT is specified, but the relationship between processability and compositional variations has yet to be established. This is in contrast with traditional engineering alloys, where acceptable ranges for solute content are routinely specified. Therefore, establishing the dependence of processing characteristics and mechanical properties on alloy composition is of critical importance. It is recognized that such correlations constitute a monumental task in alloys with multiple solutes that do not act independently. Empirical design of amorphous alloys remains the norm, particularly as it relates to mechanical behavior [refs. 15 and 35]. In the absence of composition limit specifications, QA tests must ensure that each lot of re-melt stock is amenable to producing fully-amorphous, fully-formed, and low-defect castings. Therefore, it is proposed that composition-sensitive parameters, such as GFA, viscosity, and T_x, can serve as convenient metrics of processing behavior.

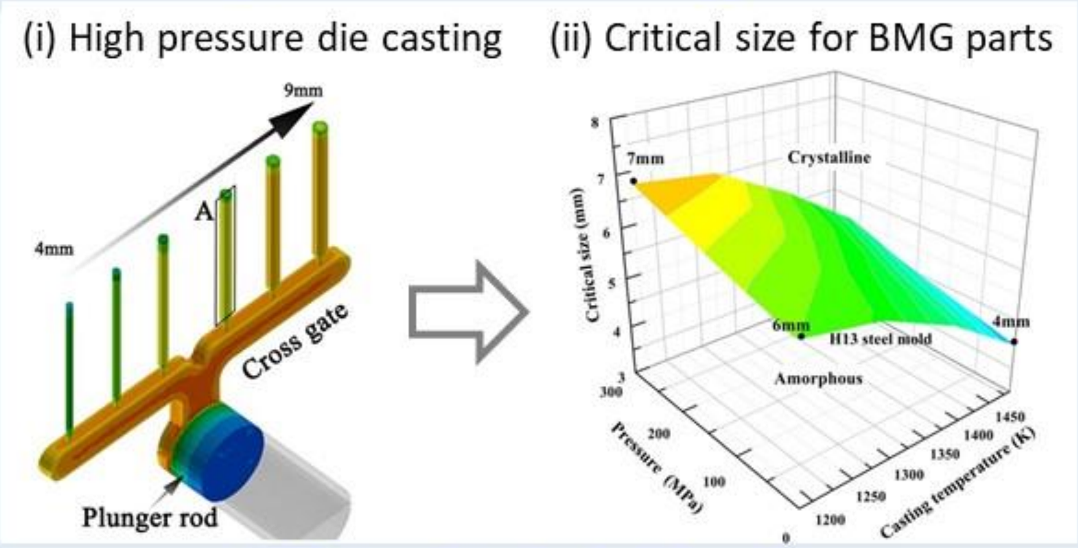
3.3. Screen Testing for Processability

Reference die designs can incorporate concepts from permanent mold or die casting of crystalline materials, and specialized methods unique to the casting of amorphous materials. The purpose of acceptance casting production is to assess the processability of the re-melt stock. Practical measures suitable for screen testing by ***Materials Suppliers*** will differ from more exacting methods suitable for formal testing by the ***Customer***. Evaluation of GFA should be the highest priority for screening tests and determining the maximum section thickness (d_{max}) that remains fully amorphous is a practical approach. Evaluation of viscosity, onset of crystallization time, and inclusion/porosity content may be classified as auxiliary options.

3.3.1. Determination of Critical Casting Thickness (GFA)

Determination of d_{max} is a common technique for assessing the castability of BMG alloys [ref. 21]. It may be defined as the maximum section thickness that retains complete amorphicity for prescribed cooling rate, casting die geometry, and heat transfer characteristics [ref. 20]. This value will be sensitive to the solute content of an alloy and may be employed as a convenient indicator of the compositional variance of ingots. Figure 12 indicates that there are two basic options for producing castings with graduated increases in section thickness for this purpose. Figure 12(a) shows a multiple rod design, and figure 12(b) shows a stepped-wedge design, both incorporating a steady increase in casting thickness [refs. 18 and 34].

(a) Multi-diameter Rod Configuration



(b) Stepped-wedge Configurations

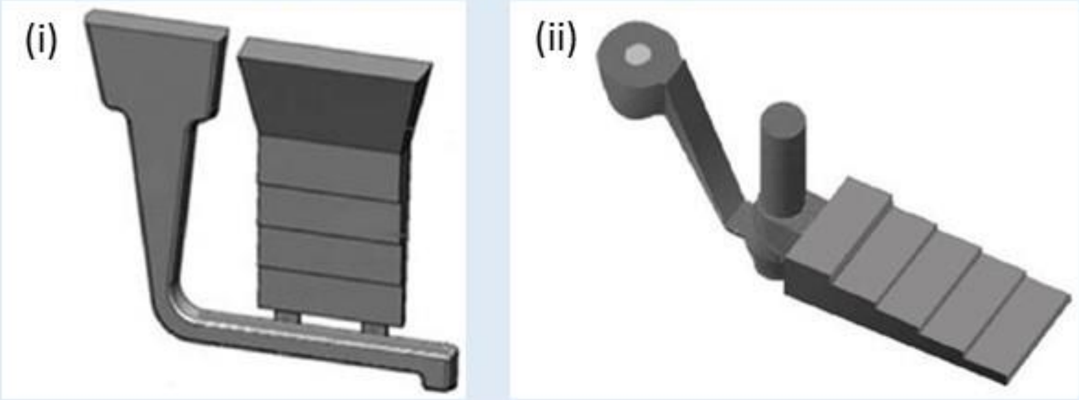


Figure 12. Design options for reference dies to determine the maximum section thickness, dmax, for producing fully-amorphous castings: (a), multi-diameter rod configuration [ref. 34]; (b), stepped-wedge configurations [ref. 18].

It is recommended that ***Visser*** designs and fabricates a reference die capable of producing a series of round bars with incrementally increasing diameter. The most recent value of d_{max} for alloy S77PT is reported as 16 mm [ref. 1]. In order to determine d_{max}, it is suggested that each casting comprise 6 bars ranging in diameter from 10 mm to 20 mm. Subsequent X-ray diffraction (XRD) analysis then determines the largest diameter in which crystal nucleation has not occurred. It is worth noting that values of d_{max} for other candidate alloys, GHDT and Vit 1, are reported as 28 mm and 22 mm, respectively [ref. 1]. This suggests that another reference die may also be required with larger, graduated diameters; e.g., 20 mm to 30 mm.

3.3.2. Determination of Fluidity (Viscosity)

Evaluating fluidity represents an optional screening test that may prove useful. This methodology determines the efficacy of metal flow into the die and has an influence on the onset of crystallization [ref. 2]. Monitoring the fluidity of molten metal is customary in foundries, but a unified test procedure is still under development [ref. 15]. A common industrial practice is to measure the distance that a specific lot of liquid metal flows in a linear channel [ref. 22]. As shown in figure 13, strip, ribbon, or spiral tests may be adapted to the current casting process and provide a practical measure of viscosity [refs. 22, 36, and 37]. The design of the strip test is most readily-adaptable to the tooling configurations common in die casting. The channels can be of varying width/thickness and valuable witness material may be provided for other screening tests.

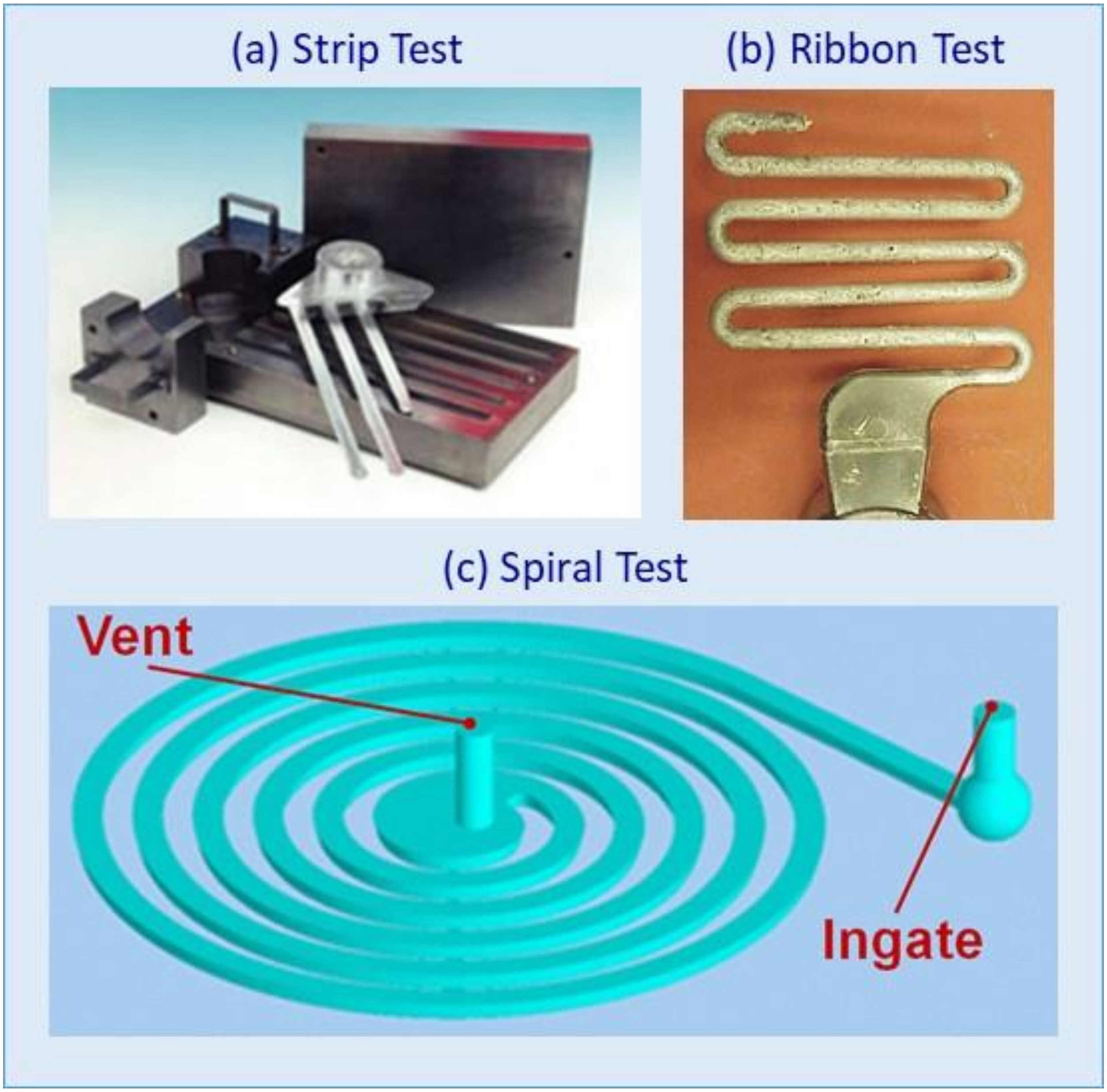


Figure 13. Design options for reference dies to determine the fluidity/viscosity for producing fully-formed castings: (a), strip test [ref. 22]; (b), ribbon test [ref. 36]; (c), spiral test [ref. 37]. Note that the strip test configuration is most readily adaptable to die casting.

3.3.3. Determination of Inclusion/Porosity Content

Evaluating inclusion and porosity content represents another optional screening test that may be of some benefit. Although such evaluation will be critical in actual parts, this may provide a measure of the susceptibility of a given lot of alloy to the formation of crystals or porosity. As shown in figure 14, there are two die configurations that are commonly used by industry; i.e., K-mold and Tatur tests [ref. 22]. These are employed to provide a statistical measurement of inclusions or gas bubbles, and porosity distribution by examination of fracture surfaces. Elements of these tests may be incorporated in to a die design that serves as an effective screening test for the current casting process.

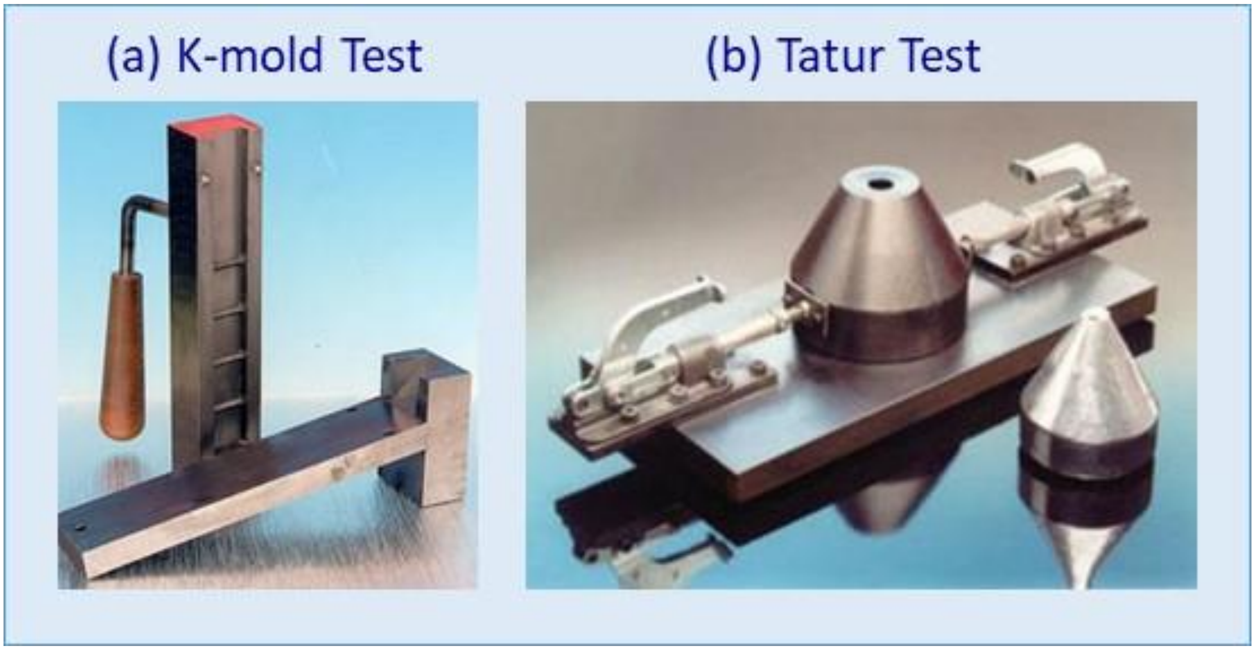


Figure 14. Design options for reference dies to determine the inclusion/porosity content for producing low-defect castings [ref. 22]: (a), the K-mold test; (b), the Tatur test. Note that the K-mold configuration is more adaptable to die casting operations.

3.4. Formal Testing for Processability

Construction of the TTT diagram for the zirconium-based BMG alloy Vitreloy 1 is illustrated in figure 15 [ref. 38]. The C curve is located by determining the onset times for isothermal crystallization as a function of undercooling temperature. In this case, a combination of differential scanning calorimetry (DSC) (blue data points) and containerless electrostatic levitation (red data points) methods have been employed to construct the curve. The “nose” of the curve represents the point at which the onset of crystallization exhibits a minimum incubation time, τ_x^*, at an undercooling temperature, T_x^*. The data for Vitreloy 1 indicate values of 60 seconds at 895K, respectively. Therefore, the location of the nose defines the critical cooling rate to avoid crystallization during solidification. The cooling profile employed for the BMG castings needs to remain within the supercooled liquid region to culminate in a fully-amorphous product. This necessity highlights the importance of constructing a TTT diagram for alloy S77PT of current vintage; e.g. oxygen content.

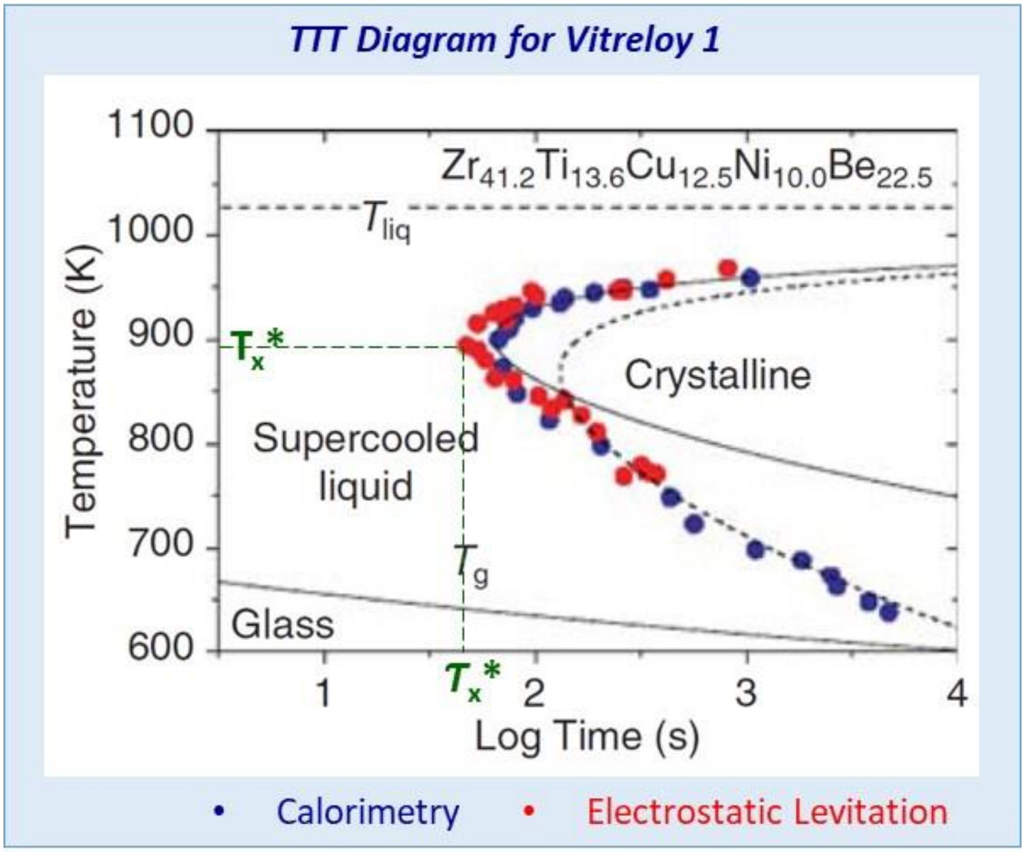


Figure 15. Construction of the TTT diagram for the zirconium-based, Vitreloy 1 [ref. 38]. The critical temperature, Tx*, and critical time, Tx*, define the minimum cooling rate for BMG castings. These thermal tolerances need to be established for current vintage S77PT.

In combination, the values of T_l, T_g, T_x, T_x*, and T_x* may be used to define the processing window for producing a fully-amorphous, fully-formed casting [ref. 20]. There is not a universal method for the containerless electrostatic levitation technique used for Vitreloy 1 above, but details of the methodology are available [ref. 39]. Standard procedures exist for differential thermal analysis (DTA), differential scanning calorimetry (DSC), thermo-mechanical analysis (TMA), and X-ray diffraction (XRD) methodologies. Typically, data relevant to TTT diagram construction can be collected in a variety of operating modes, such as isothermal holding, varying heating rate, and varying cooling rate. The traditional analytical methods may draw on the following guidelines:

ASTM E794; Standard Test Method for Melting And Crystallization Temperatures By Thermal Analysis [ref. 40]

ASTM E1356; Standard Test Method for Assignment of the Glass Transition Temperatures by Differential Scanning Calorimetry [ref. 40]

ASTM E1545; Standard Test Method for Assignment of the Glass Transition Temperature by Thermomechanical Analysis [ref. 40]

As a reference, representative data for alloy S77PT are available in the literature [ref. 10]. For evaluation of GFA, typical signatures produced via DTA and DSC methods are shown in figure 16. For evaluation of viscosity and amorphicity, typical signatures produced via TMA and XRD methods are shown in **Error! Reference source not found.**.

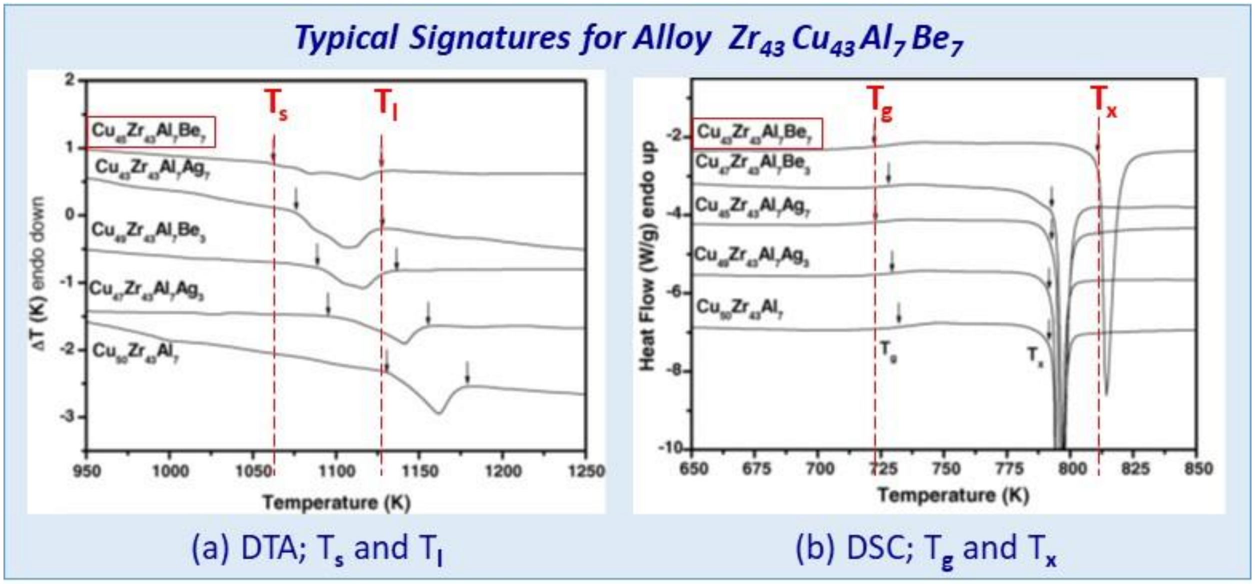


Figure 16. Representative analytical data for alloy S77PT: (a), DTA to determine the melting temperature range (Tl); (b), DSC to determine the glass transition and crystallization temperatures (Tg and Tx) [ref. 10].

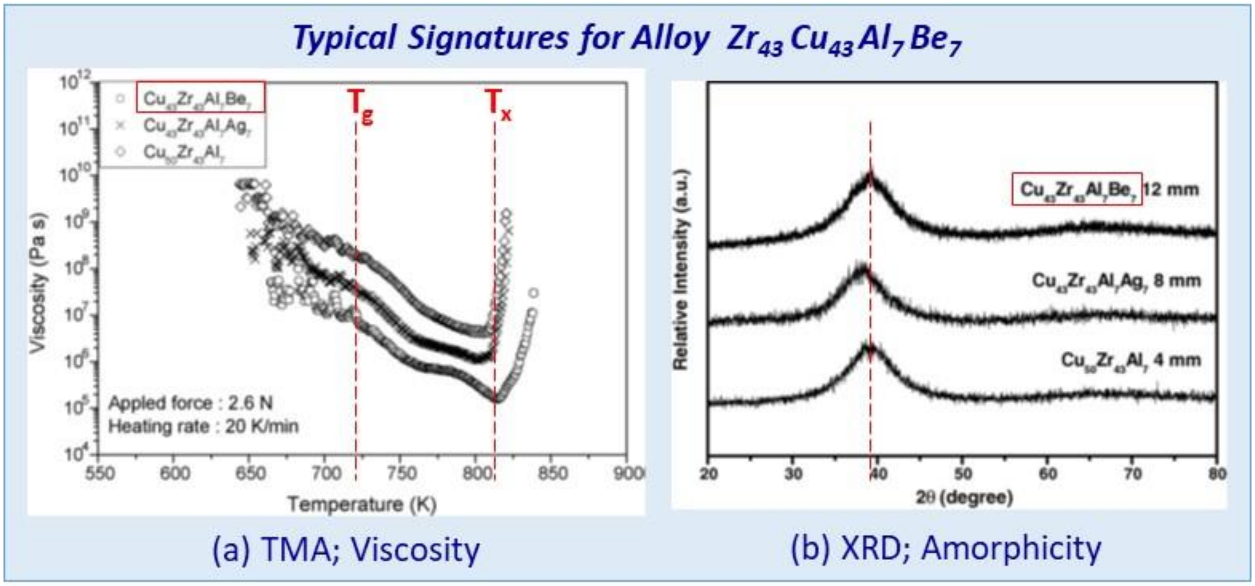


Figure 17. Representative analytical data for alloy S77PT: (a), TMA to determine viscosity, the inverse of fluidity; (b), XRD to determine the relative crystallinity or amorphicity [ref. 10].

3.5. Performance Testing of Acceptance Castings

The ***Customer*** needs to perform an oversight role during the development phase of BMG gear manufacturing. An important function of the data gathered is to define composition limits by correlating variations with processability and performance. Such specifications will lead to maturation of the materials supply chain and a reduction in such process monitoring. Implementation of the concept of producing a suite of round bars for determination of d_{max} will create a convenient opportunity for physical property testing. Witness materials will also be generated with dimensions highly suitable for further mechanical property testing, although some post-cast machining may be required. The need to produce round bar configurations within the qualification castings destined for performance evaluation will be reduced. Consequently, fully-amorphous round bars produced during "critical casting thickness" trials will be available for the following formal tests:

- Physical properties

  ASTM B311; Standard Test Method for Density of Powder Metallurgy (PM) Materials Containing Less Than Two Percent Porosity [ref. 41]

  ASTM E1225; Standard Test Method for Thermal Conductivity of Solids Using the Guarded-Comparative-Longitudinal Heat Flow Technique [ref. 40]

  ASTM E228; Standard Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer [ref. 40]

- Mechanical properties

  ASTM E9; Standard Test Methods of Compression Testing of Metallic Materials at Room Temperature (modulus, strength) [ref. 26]

  ASTM E143; Standard Test Method for Shear Modulus at Room Temperature [ref. 26]

  ASTM E1450; Standard Test Method for Tension Testing of Structural Alloys in Liquid Helium (modulus, strength, ductility) [ref. 26]

4. Performance Testing

4.1. Qualification Castings for Performance Evaluation

4.1.1. Reference Die Geometry

The role of qualification castings is dedicated to the mechanical behavior of the amorphous castings produced by ***Visser***. It is suggested that a reference die containing a series of cavities with geometries conforming with ASTM specifications is designed and fabricated. As illustrated in figure 18(a) and 18(b), incumbent die casting techniques can produce multi-configuration specimens concurrently; e.g., flat, square, round, and/or plate blanks [ref. 18]. The close tolerances afforded by the process also allow casting of smooth and notched specimen configurations. For example, ***Visser*** might produce multiples of specimens to E8 and E399 dimensions to fulfill a vital function of formal testing by the ***Customer***.

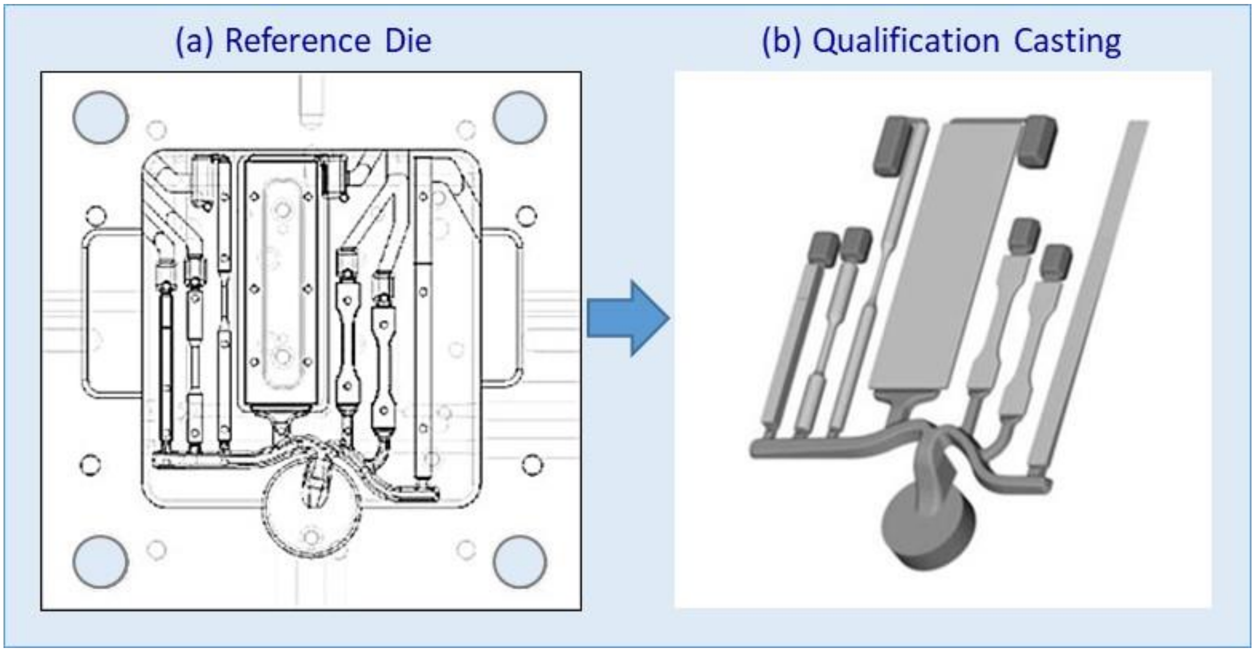


Figure 18. Production of witness materials to ASTM dimensions for performance testing: (a), configuration of multi-cavity reference die; (b), qualification casting consisting of a group of test specimens [ref. 18].

Guidance on effective die designs for producing mechanical test specimens may be obtained from other industries that employ injection molding [ref. 42]. Practices established for casting of thermoplastic metals and polymers to standardized dimensions include

> ASTM B883; Standard Specification for Metal Injection Molded (MIM) Materials [ref. 41];

ASTM B969; Standard Specification for Aluminum-Alloy Castings Produced by Squeeze Casting, Thixocast and Rheocast Semi-Solid Casting Processes [ref. 43];

ASTM D3641; Standard Practice for Injection Molding Test Specimens of Thermoplastic Molding and Extrusion Materials [ref. 44];

ISO 294; Plastics — Injection moulding of test specimens of thermoplastic materials [ref. 45]; and

ISO 20753; Plastics — Test specimens [ref. 46].

Figure 19 shows examples of standards for sprue, runner and gate geometries employed in the plastics industry. Examples of multi-cavity dies for creating specimen geometries suitable for bend or tension testing are illustrated in figure 19(a) and 19(b) [ref. 44]. In addition, a multi-cavity die compatible with producing a mixture of test specimens is shown figure 19(c) [ref. 45]. The configurations highlight the need for molten material to feed into the grip section, as opposed to the gage section, of all test specimens. This is important for notch-sensitive BMG castings, because the potential to introduce flaws during post-cast machining of the test section is eliminated.

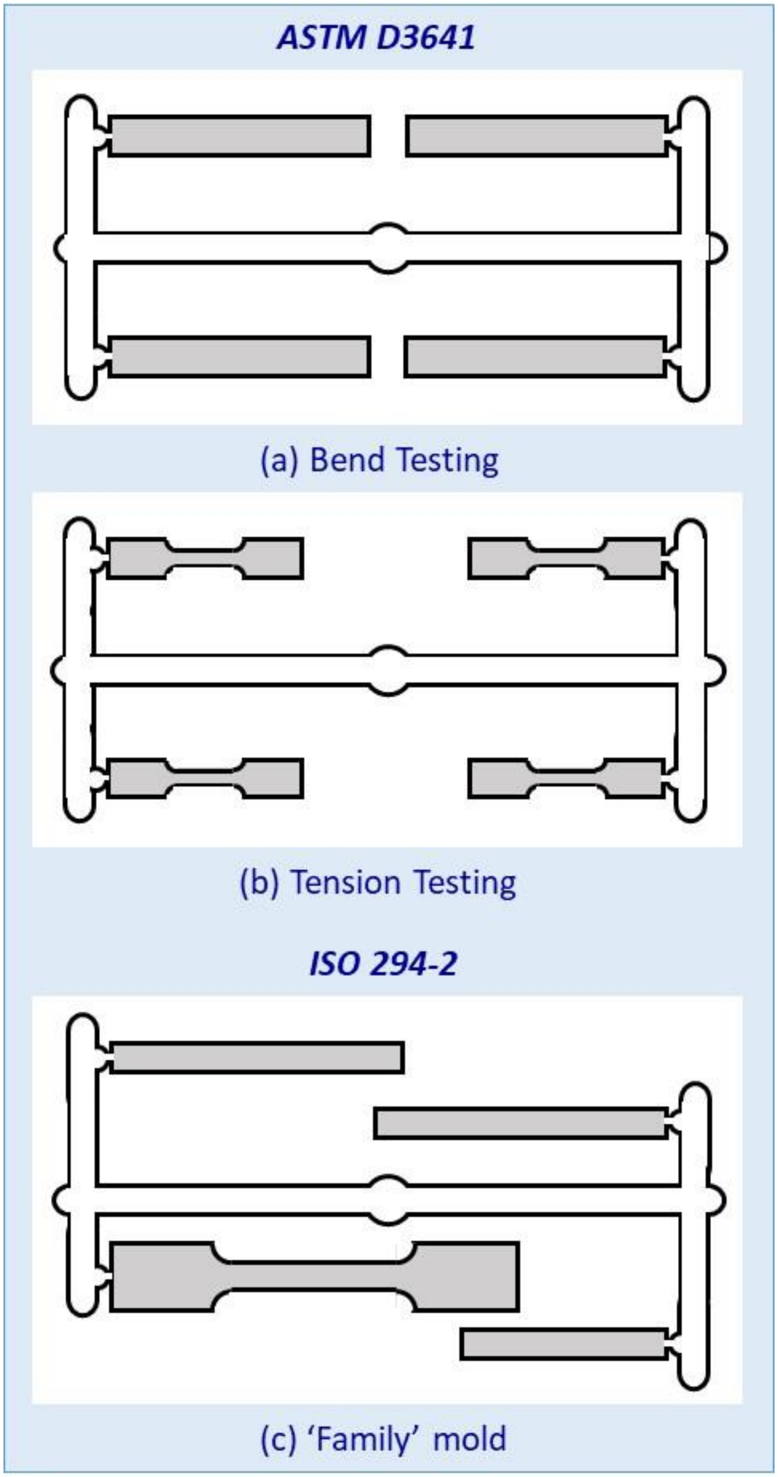


Figure 19. Die configurations used for casting of test specimens in the plastics industry: (a), bend testing [ref. 44]; (b), tension testing [ref. 44]; (c), "family" mold [ref. 45]. Note that an important feature is that molten material feeds into the grip section, rather than the gage section, in all cases.

4.1.2. Die Cavity Geometries

Decisions will need to be made on the configuration of multi-cavity reference dies for producing qualification castings. In its simplest form, a die may include multiples of a single type of test specimen. In a more practical configuration, a die may comprise a variety of test specimen dimensions. The key aspect will be producing test specimens of comparable thickness to the BMG gear components. The testing requirements include monotonic loading only; i.e., no cyclic (fatigue), or extended duration (wear) tests. In addition, gathering of both ambient and cryogenic temperature data will be required for most mechanical properties. Depending on the type of data to be generated, specimen blank configurations may be selected from the following:

- Smooth flat bars for tension and bend testing
- Smooth round bars for compression and torsion testing
- Notched square bars for impact and bend testing
- Notched flat plates for tensile (toughness) testing

4.2. Screen Testing for Performance

It is suggested that screening tests performed by ***Materials Suppliers*** be confined to smooth flat bar and notched square bar configurations. The mechanical testing proposed involves readily-available methods and fixtures with unified test procedures.

4.2.1. Smooth Flat Bars

Screen testing procedures may be selected from

> ASTM C1161; Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature [ref. 47];
>
> ASTM E290; Standard Test Methods for Bend Testing of Material for Ductility [ref. 26]; or
>
> ASTM E855; Standard Test Methods for Bend Testing of Metallic Flat Materials for Spring Applications Involving Static Loading [ref. 26].

Examples of semi-articulated test fixtures, conforming with ASTM C1161 procedures, are shown in figure 20 [ref. 47]. The choice of either 3-point or 4-point bending is presented because the brittleness of the BMGs to be tested is uncertain. The decision may ultimately hinge on compatibility with subsequent formal testing to be conducted at cryogenic temperatures. In 3-point flexural testing, the maximum stress is confined directly below the central bearing load, figure 20(a). In 4-point flexural testing, the maximum stress is dissipated over a larger portion of the gage section, figure 20(b). The effect of flaws on crack initiation is less severe, which improves the accuracy of data gathered from notch-sensitive materials. The standard specifies that the loading span for 3-point and 4-point bending tests can be 20, 40 or 80 mm, all of which are compatible with typical BMG gear dimensions.

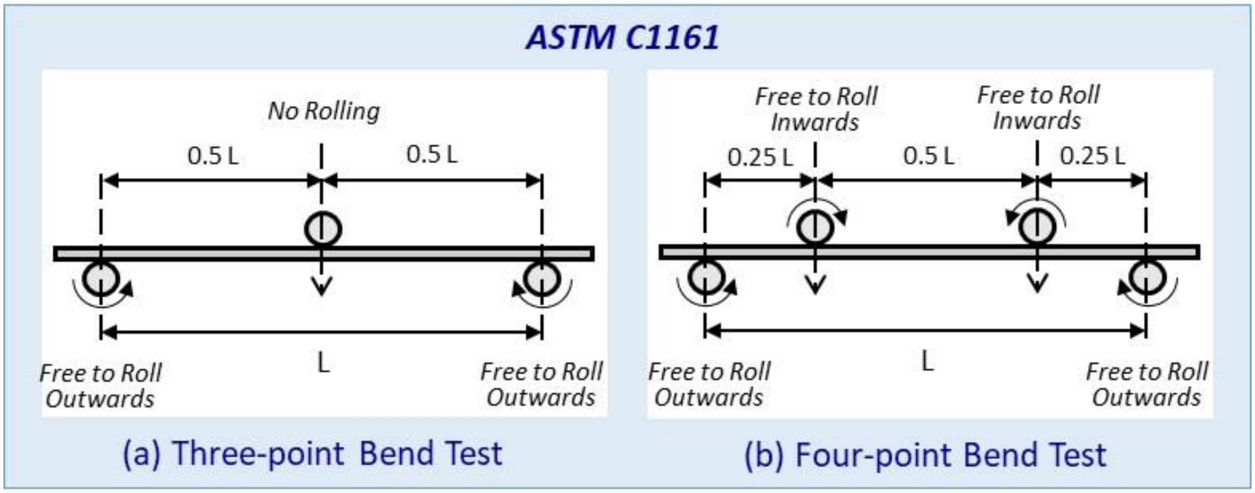


Figure 20. Flexural tests incorporating semi-articulated load fixtures: (a), 3-point bend testing; (b), 4-point-¼ point bend testing [ref. 47]. Note that 3-point and 4-point configurations tend to be applied to ductile and brittle materials, respectively.

4.2.2. Notched Square Bars

This specimen configuration is more applicable to planetary gears than strain wave gears due to section thickness compatibility. Casting of pre-notched specimens could be a definite advantage in this case. Screen testing procedures may be selected from

> ASTM E23; Standard Test Methods for Notched Bar Impact Testing of Metallic Materials [ref. 26]; or
>
> ASTM E2248; Standard Test Method for Impact Testing of Miniaturized Charpy V-Notch Specimens [ref. 26].

Figure 21(a) and 21(b), shows an Izod impact test specimen in accordance with E23 and a miniaturized Charpy impact test specimen in accordance with E2248 [ref. 26]. The dimensions of the standard V-notch Izod test specimens are 75 mm L x 10 mm W x 10 mm T. The dimensions of the standard V-notch and U-notch Charpy test specimens are 55 mm L x 10 mm W x 10 mm T. There are unnotched versions of the standard Izod and Charpy tests that are customarily employed for powder metallurgy structural materials that conform with the same, respective dimensions. The dimensions of the miniaturized Charpy test specimens are 24.13 mm L x 4.83 mm W x 4.83 mm T, which may be the most appropriate for BMG gears.

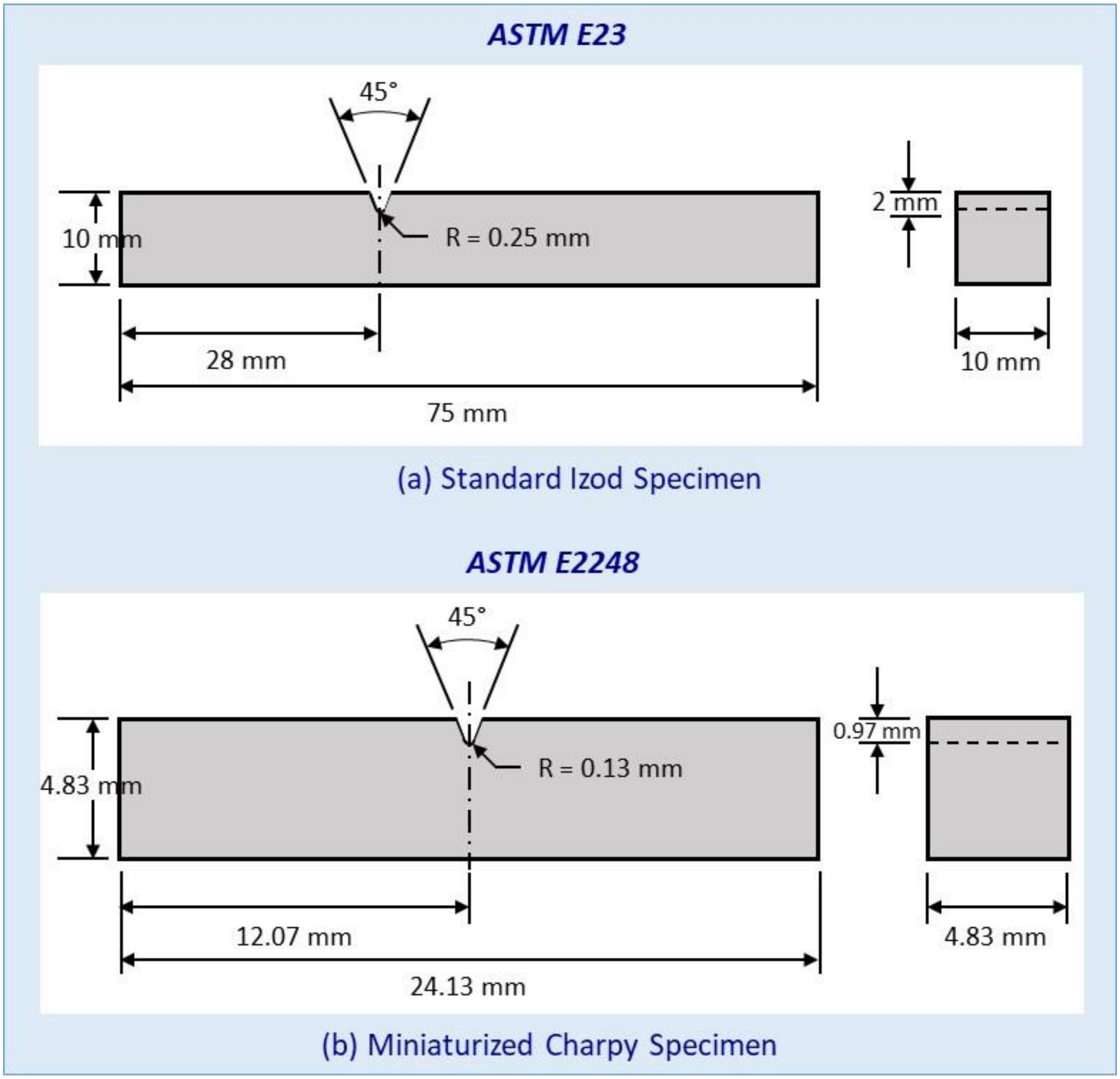


Figure 21. Standard specimen configurations for impact testing [ref. 26]: (a), E23 - standard Izod specimen; (b), E2248 - miniaturized Charpy specimen. Note that this type of test may be more applicable to planetary gears due to section thickness compatibility.

4.3. Formal Testing for Performance

It is suggested that formal tests conducted by the ***Customer*** be expanded to include smooth round bar and notched flat plate configurations. The mechanical testing proposed involves complex specimen preparation or set-up, and specialized test equipment. Formal testing procedures may be selected from

ASTM E8; Standard Test Methods for Tension Testing of Metallic Materials (modulus, strength, ductility) [ref. 26]; or

ASTM E132; Standard Test Method for Poisson’s Ratio at Room Temperature [ref. 26].

4.3.1. Smooth Flat and Round Bars

As shown in figure 22, tensile testing in accordance with E8 includes specimen configurations using both flat and round bar stock [ref. 26]. There is a selection of dimensions for the rectangular tension test specimen shown in figure 22(a). Standard sheet-type specimens are 200 mm L x 20 mm W x thickness of the material. Standard subsize specimens are 100 mm L x 10 mm W x thickness of the material. The smaller configuration is ideal for evaluating the tensile properties of BMG gears, but the larger configuration is more compatible with measuring Poisson’s ratio.

There is a broad spectrum of dimensions for the round tension test specimen shown in figure 22(b). The standard specimen has reduced section dimensions of 56 mm L x 12.5 mm D. The smallest subsize specimen of the four options listed has reduced section dimensions of 16 mm L x 2.5 mm D. Therefore, the specimen configuration may be chosen according to the BMG gear type and size. This specimen configuration is also more compatible with common fixturing employed for cryogenic testing [ref. 29]. Formal procedures for such tests may be selected from

ASTM E1450; Standard Test Method for Tension Testing of Structural Alloys in Liquid Helium (modulus, strength, ductility) [ref. 26];

ISO 6892; Metallic materials — Tensile testing — Part 3: Method of test at low temperature [ref. 48]; or

ISO 15579; Metallic materials — Tensile testing at low temperature [ref. 49].

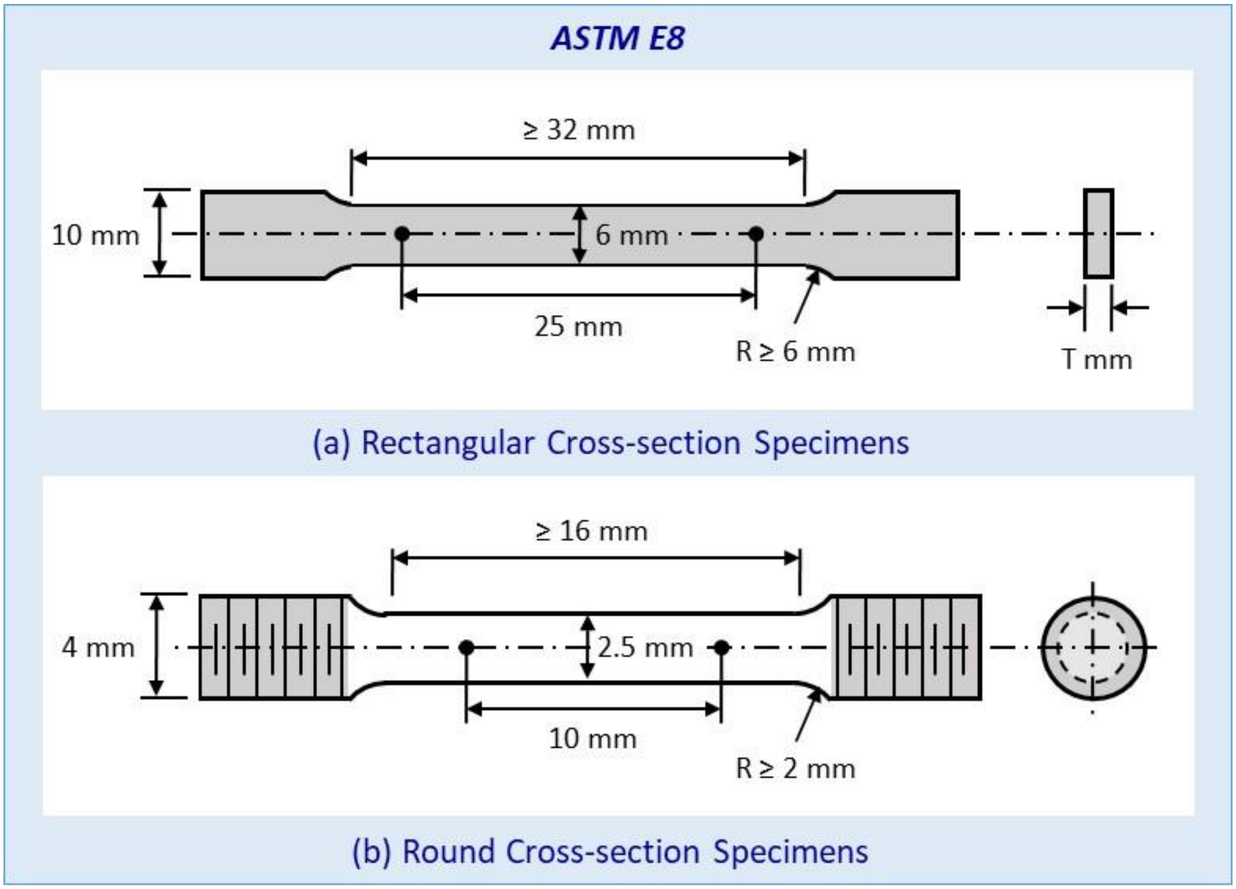


Figure 22. Standard specimen configurations for E8 tension testing [ref. 26]: (a), specimens with rectangular cross-section; (b), specimens with round cross-section. Note that the subsize versions are more compatible with BMG gear dimensions.

The E8 specimen configurations shown in figure 23 are included because they are customarily employed for many types of castings. Figure 23(a) represents the standard test specimen used for die castings and typical dimensions are 230 mm L x 10 mm D. Figure 23(b) represents the standard test specimen used for malleable iron and typical dimensions are 190 mm L x 16 mm D. The section thicknesses are compatible, but subsize versions are not listed for either of these specimen configurations in terms of length. Figure 23(c) represents the standard test specimen used for cast iron that constitutes the most adaptable configuration for BMG gears. There is a choice of specimen sizes, the smallest dimensions currently available comprise 93 mm L x 20 mm D. In all cases, the overall dimensions quoted are approximate, because the length of the grip section is flexible.

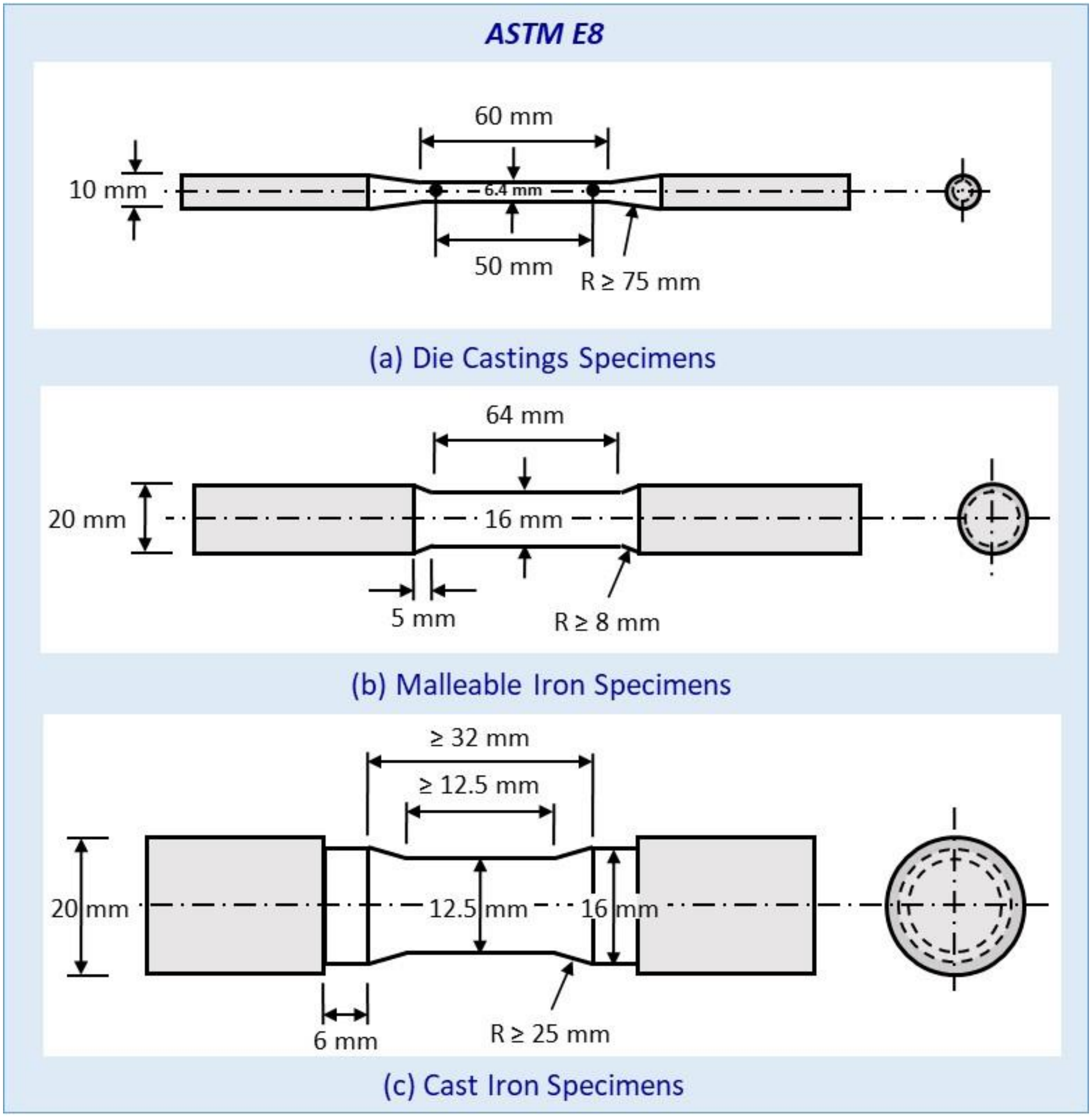


Figure 23. Standard specimen configurations for E8 tension testing of castings [ref. 26]: (a), specimens from die castings; (b), specimens from malleable iron; (c), specimens from cast iron.

4.3.2. Notched Flat Plates

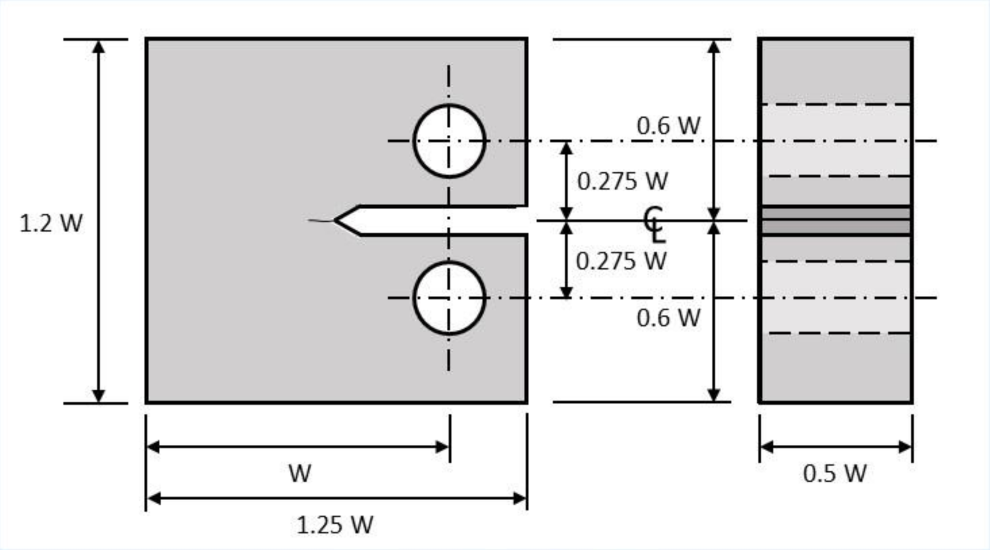
Fracture toughness testing represents the traditional method for assessing the resistance to crack formation and growth in metallic materials and data may correlate with fatigue behavior [ref. 4]. Although the few standards pertaining to evaluation of toughness are highly-focused, each of the standards covers a very broad range of test methods. The choice of test depends on the relationship between the toughness and thickness of the material. The majority of amorphous alloys are classified as quasi-brittle materials, but zirconium-based alloys exhibit fracture toughness values comparable to crystalline structural alloys [ref. 50]. The standard procedures for formal testing are outlined in

> ASTM E399; Standard Test Method for Linear-Elastic Plane-Strain Fracture Toughness K_{Ic} of Metallic Materials [ref. 26]; and
>
> ASTM E1820; Standard Test Method for Measurement of Fracture Toughness [ref. 26].

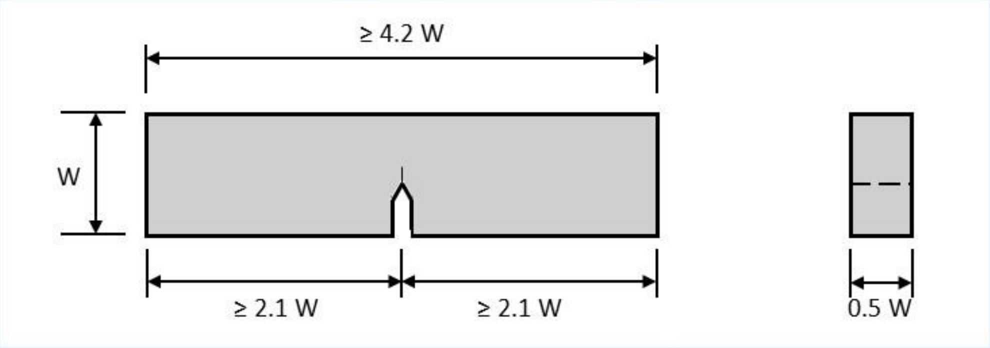
The most common options include compact tension (CT) and single edge notch bend (SENB) tests, as shown in figure 24 [ref. 26]. Both types of specimen contain a notch that is sharpened with fatigue pre-cracks for accurate data. The specimen configurations are displayed as proportions, rather than actual dimensions, and accurate toughness data is dependent on specimen size. Specimen dimensions are flexible for each method, but a minimum thickness of 1.6 mm is specified for valid data. Experience suggests that CT specimen dimensions corresponding to W = 25 mm represent the minimum size that can accommodate the necessary instrumentation. This dictates that the specimen thickness (T=W/2) is 12.5 mm for plane-strain fracture toughness measurements.

Evaluation of amorphous alloys frequently employs SENB testing and representative specimen dimensions comprise 36 mm L x 8 mm W x 2.1 mm T [ref. 50], and 26 mm L x 6 mm W x 3 mm T [ref. 51]. Obviously, data compiled in accordance with either of these standards is acceptable, but preliminary tests will be required to determine the best approach. This will include consideration of a common method for both ambient and cryogenic temperature testing of gear materials. A unified test will permit the damage tolerance of candidate BMG alloys to be ranked, even if specimen dimensions have to deviate from E399 specifications. Ultimately, the choice of test procedure will govern the design of the reference die; i.e., the thickness of qualification castings.

ASTM E399/E1820



(a) Compact Tension (CT)



(b) Single Edge Notch Bend (SENB)

Figure 24. Most common specimen configurations for E399/E1820 fracture toughness tests [ref. 26]: (a), compact tension (CT) testing; (b), single edge notch bend (SENB) testing.

4.3.3. Notched Square Bars

It should be noted that this specimen configuration is not the best choice for flexspline gears, with a maximum thickness of 2 mm. Square bars for both impact and bending toughness tests have a section thickness of ~ 5 mm, which is more compatible with planetary gears. Formal testing procedures may be selected from

> ASTM E23; Standard Test Methods for Notched Bar Impact Testing of Metallic Materials [ref. 26];
>
> ASTM E1820; Standard Test Method for Measurement of Fracture Toughness [ref. 26];
>
> ASTM E2248; Standard Test Method for Impact Testing of Miniaturized Charpy V-Notch Specimens [ref. 26]; and

ASTM E2899; Standard Test Method for Measurement of Initiation Toughness in Surface Cracks Under Tension and Bending [ref. 26].

4.4. Baseline QA Testing of BMG Gear Castings

There is a wide selection of macro-hardness tests that are applicable to preliminary evaluation of BMG gears. Ideally, the choice of a single method across-the-board would be desirable for comparison purposes. Micro-hardness testing of cross-sections may also prove beneficial, if vendors have the capability.

4.4.1. Screen Testing (*Visser*)

Hardness testing represents the traditional method for assessing the pedigree of metallic materials and data may correlate with strength, ductility and/or wear resistance [ref. 4]. Conventional macro- and micro-hardness testing procedures may be selected from the following:

- Macro-hardness

  ASTM E10; Standard Test Method for Brinell Hardness of Metallic Materials [ref. 26]

  ASTM E18; Standard Test Methods for Rockwell Hardness of Metallic Materials [ref. 26]

  ASTM E92; Standard Test Methods for Vickers Hardness and Knoop Hardness of Metallic Materials [ref. 26]

- Data conversion

  ASTM E140; Standard Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Superficial Hardness, Knoop Hardness, Scleroscope Hardness, and Leeb Hardness [ref. 26]

- Micro-hardness

  ASTM E384; Standard Test Method for Microindentation Hardness of Materials [ref. 26]

Among the most-widely-used macro-hardness techniques, the Brinell method uses a steel ball as an indenter and is used almost exclusively for evaluating castings. The Rockwell (C scale) method uses a diamond cone as an indenter and is frequently employed for QA of wrought products. From the perspective of micro-hardness, the Knoop method uses an elongated diamond pyramid as an indenter and is employed when closely-spaced data are required. The Vickers method uses a tetragonal diamond pyramid as an indenter and is a widely used, multi-purpose technique. An advantage of this method is the load range capability, which extends from the micro- to the macro-hardness regime. The difference between the indenter geometry for micro-hardness testing on the Knoop or the Vickers scale is illustrated in figure 25(a) and 25(b) [ref. 26]. The Knoop method is a good choice for evaluating small parts and thin sections because of the ability to get into tight spaces. However, the Vickers indenter is better-suited to isotropic materials, such as amorphous alloys. Therefore, both techniques offer advantages for assessment of BMG gears.

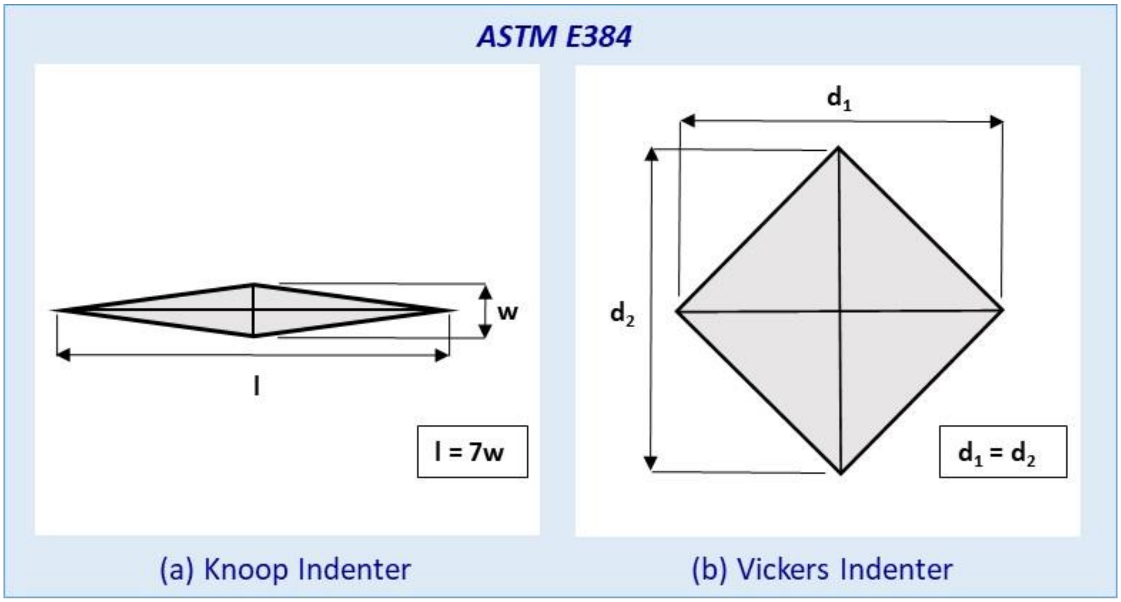


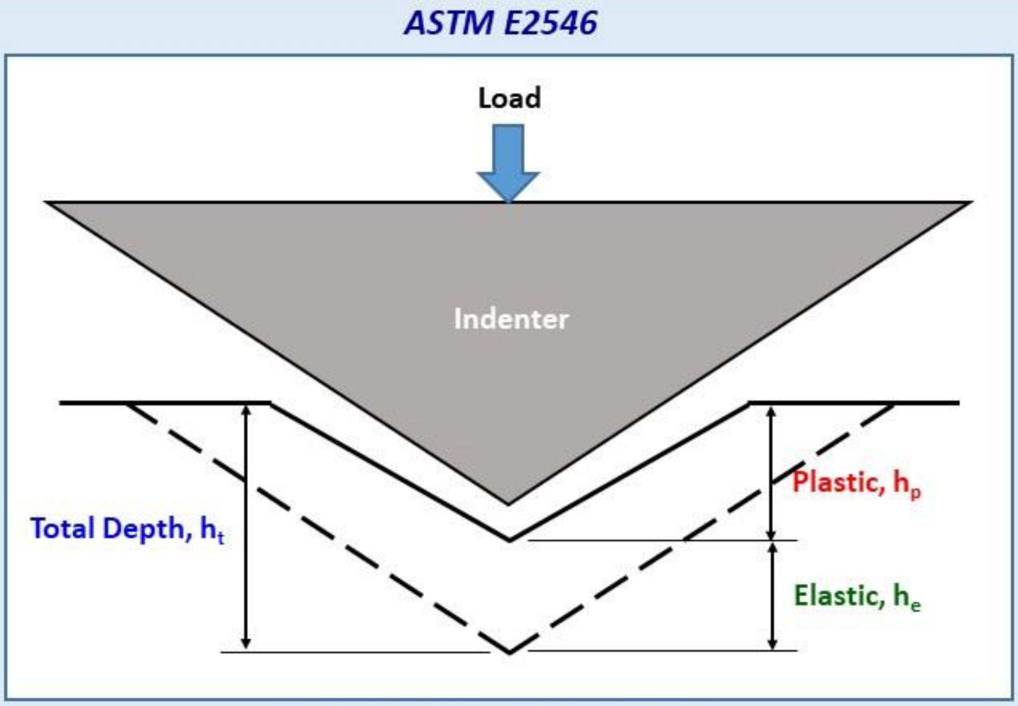
Figure 25. Selection of standard micro-hardness testing procedures [ref. 26]: (a), geometry of Knoop indenter; (b) geometry of Vickers indenter. Note that the Knoop method is advantageous for tight spaces, and the Vickers method is beneficial for isotropic materials.

4.4.2. Formal Testing (*Customer*)

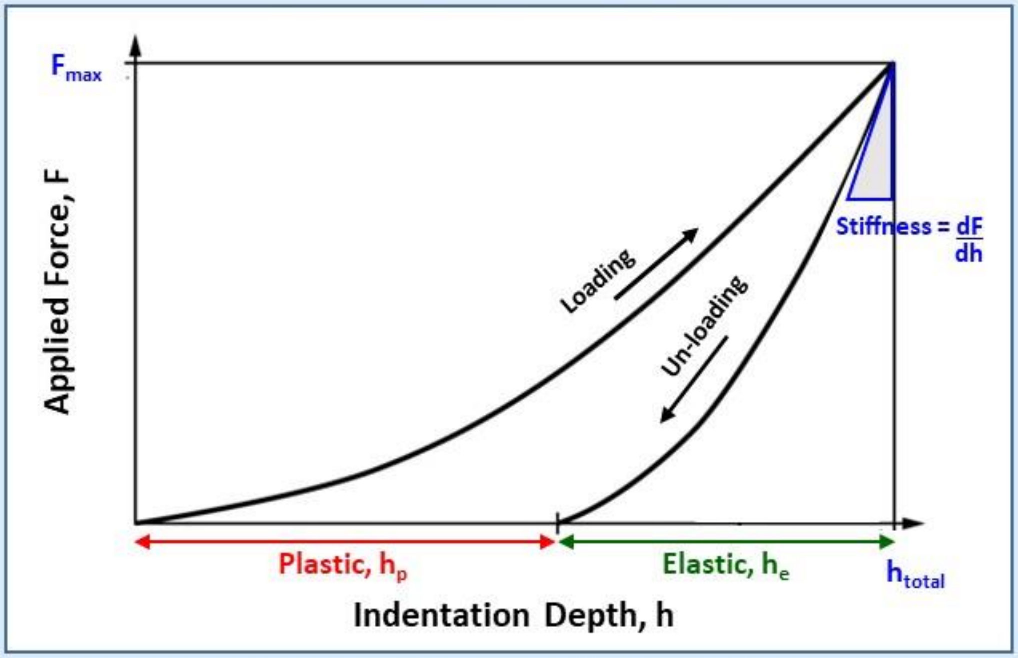
Micro- and nano-hardness testing can also be employed to provide a measure of elastic modulus and fracture toughness, but dedicated equipment is required [ref. 4]. Instrumented and specialized indentation testing procedures may be selected from

> ASTM E2546; Standard Practice for Instrumented Indentation Testing [ref. 26];
>
> ISO 14577; Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method [ref. 53]; and
>
> ISO 28079; Hardmetals — Palmqvist toughness test [ref. 54].

Details on the procedure for instrumented indentation testing (IIT) in accordance with E2546 are outlined in figure 26 [ref. 26]. IIT permits the load and depth of the indentation to be measured simultaneously, leading to the construction of a load-depth curve. The indenter is inserted and withdrawn at prescribed rates, such that the material/indenter interaction provides a measure of hardness and stiffness. The slope of the unloading curve at maximum load is used to calculate the elastic modulus. In addition, the indentation fracture toughness using a specialized method can be estimated, if the material is sufficiently brittle. Typically known as Palmqvist toughness, the technique involves measurement of the lengths of the cracks created at the tips of the indentation, as shown in figure 27(a) [ref. 54]. Details on the procedure for conducting indentation toughness tests in accordance with ISO 28079 are outlined in figure 27(b) [ref. 55]. Although such standardized procedures exist, the utility of the data for quantifying the fracture toughness of brittle materials remains uncertain [ref. 56].

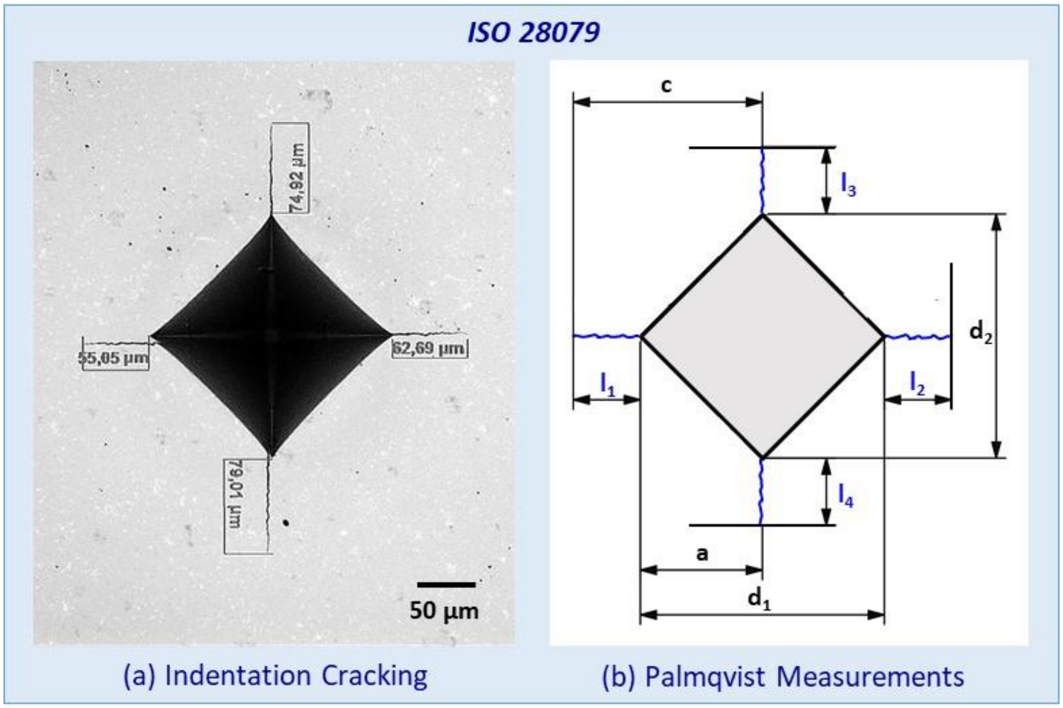


(a) Indentation Cross-section



(b) Loading/Measurement Procedure

Figure 26. Instrumented indentation testing to determine hardness and estimate stiffness [ref. 26]: (a), indentation cross-section; (b) loading/measurement procedure.



(a) Indentation Cracking

(b) Palmqvist Measurements

Figure 27. Specialized indentation testing to estimate fracture toughness: (a), indentation cracking [ref. 54]; (b) Palmqvist measurements [ref. 55]. Note that the accuracy of this methodology is debatable for brittle materials.

4.5. QA Testing Specific to Flexspline Gears

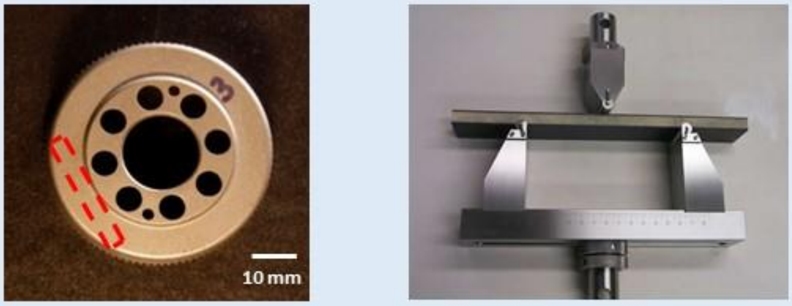
4.5.1. Candidate Screen Tests (*Visser*)

Flexspline components for SWGs are small, complex-shaped, thin-walled castings. Screen testing may be defined as any method by which a material or process can be validated quickly and cheaply. Therefore, specimen preparation needs to include simple, metallographic-type procedures and exclude post-cast machining. Mechanical properties such as bending strength and hoop strength of simple sections are suggested as potential metrics for the screening tests. As a result, a number of innovative tests have been identified that can be applied to flexspline gears, shown in figure 28. The testing methods have been adapted from ASTM standards formulated for other materials and structures; e.g., composites. In addition to conducting 3-point bend testing in accordance with ASTM C1161 [ref. 26], it is proposed that test procedures may be selected from

> ASTM D2344; Standard Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates [ref. 57];
>
> ASTM D6415; Standard Test Method for Measuring the Curved Beam Strength of a Fiber-Reinforced Polymer-Matrix Composite [ref. 57]; and
>
> ASTM D2290; Standard Test Method for Apparent Hoop Tensile Strength of Plastic or Reinforced Plastic Pipe [ref. 58].

(a) Flat sections

ASTM C1161



(b) Curved sections

ASTM D2344

(c) Right-angled Sections

ASTM D6415

(d) Circular Sections

ASTM D2290

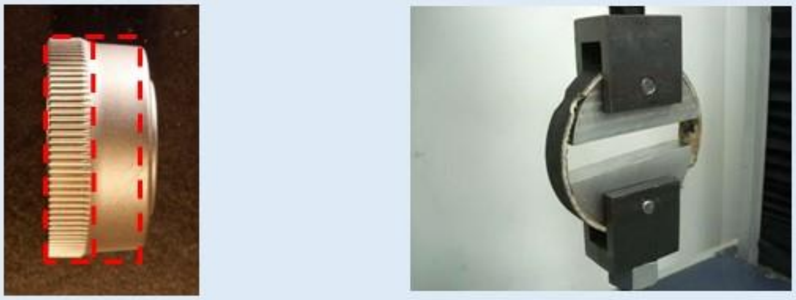


Figure 28. Screen testing of flexspline gears: (a), flat sections, using 3-point bend tests [ref. 47]; (b), curved sections, using 3-point bend tests [ref. 57]; (c), right-angled sections, using 4-point bend tests [ref. 57]; (d), circular sections, using split-disk tests [ref. 58].

Flat sections provide material suitable for generating planar macro-hardness profiles in accordance with E10, E18, or E92 [ref. 26]. Likewise, flat and curved sections provide material suitable for generating cross-sectional micro-hardness profiles in accordance with E384 [ref. 26]. Bending strength can be determined by 3-point bend testing of flat sections in accordance with C1161 [ref. 47], or curved sections in accordance with D2344 [ref. 57]. Bending strength can also be determined by 4-point bend testing of right-angle sections in accordance with D6415 [ref. 57]. Hoop strength can be evaluated by split-disk testing of circular sections in accordance with D2290 [ref. 58]. There is an opportunity to extract hoop specimens from the smooth section or the toothed section of the flexspline. A comparison of the results might also provide some measure of the notch sensitivity of the BMG casting.

Non-destructive evaluation (NDE) of porosity and inclusions in BMG gear castings is essential, but details are not covered in this report. However, a preliminary scan for defects (inclusions, porosity) on whole components may also be a feasible screening test. Depending on equipment availability, candidate NDE procedures may include, but are not restricted to

> ASTM E243; Standard Practice for Electromagnetic (Eddy Current) Examination of Copper and Copper-Alloy Tubes [ref. 59];
>
> ASTM E1030; Standard Practice for Radiographic Examination of Metallic <u>Castings</u> [ref. 59];
>
> ASTM E1734; Standard Practice for Radioscopic Examination of <u>Castings</u> [ref. 59];
>
> ASTM E1932; Standard Guide for Acoustic Emission Examination of Small Parts [ref. 59]; and
>
> ASTM E2001; Standard Guide for Resonant Ultrasound Spectroscopy for Defect Detection in Both Metallic and Non-metallic Parts [ref. 59].

4.5.2. Candidate Formal Tests (*Customer*)

4.5.2.1. Sectioned Components

Specimen extraction and sizing to appropriate dimensions must involve minimal machining operations to reduce the chance of damage. Samples for evaluating physical properties important to the gear application, and limited mechanical properties, may be prepared from flat or curved sections. Formal testing procedures for evaluating thermal properties may be selected from

> ASTM E228; Standard Test Method for <u>Linear Thermal Expansion</u> of Solid Materials With a Push-Rod Dilatometer [ref. 40];
>
> ASTM E831; Standard Test Method for <u>Linear Thermal Expansion</u> of Solid Materials by Thermomechanical Analysis [ref. 40];
>
> ASTM E1225; Standard Test Method for <u>Thermal Conductivity</u> of Solids Using the Guarded-Comparative-Longitudinal Heat Flow Technique [ref. 40]; and
>
> ASTM E1952; Standard Test Method for <u>Thermal Conductivity</u> and Thermal Diffusivity by Modulated Temperature Differential Scanning Calorimetry [ref. 40].

Dynamic mechanical analysis (DMA) is a technique that may prove convenient for evaluating flexspline gear materials. The apparatus can be operated in dynamic (resonance) mode or static mode [ref. 60]. The potential benefit stems from the ability to readily determine dynamic Young's modulus, shear modulus, and Poisson's ratio as a function of temperature. Customarily applied to polymeric materials, metallic materials can be analyzed in dynamic mode if caution is exercised during measurements. Thin test sections need to be employed, such that the stiffness of the metal specimen is much lower than the stiffness of the metal test fixturing. The technique has already been applied to analysis of the deformation behavior of zirconium-based BMGs [ref. 61]. Formal testing procedures for dynamic tests using DMA may be selected from

> ASTM C623; Standard Test Method for Young's Modulus, Shear Modulus, and Poisson's Ratio for Glass and Glass-Ceramics by Resonance [ref. 62]; and
>
> ASTM E1875; Standard Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio by Sonic Resonance [ref. 26].

As illustrated in figure 29, operating the DMA in static mode may be one of the few ways way to perform mechanical testing on fabricated components. For example, 3-point bend tests may provide a formal measure of cryogenic modulus that can be correlated with results from witness materials testing. ASTM/ISO standards for bend testing of metallic materials at cryogenic temperatures are unavailable. A recurring problem is definition of the span width when accounting for thermal contraction. Existing standards for 3-point flexural testing of other materials that may provide some guidance include ASTM D790 and ISO 178. Consequently, formal testing procedures for static tests using DMA may employ the following guidelines:

> ASTM C1161; Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature [ref. 47]
>
> ASTM D790; Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials [ref. 63]
>
> ASTM E290; Standard Test Methods for Bend Testing of Material for Ductility [ref. 26]
>
> ASTM E855; Standard Test Methods for Bend Testing of Metallic Flat Materials for Spring Applications Involving Static Loading [ref. 26]
>
> ISO 178; Plastics — Determination of flexural properties [ref. 64]

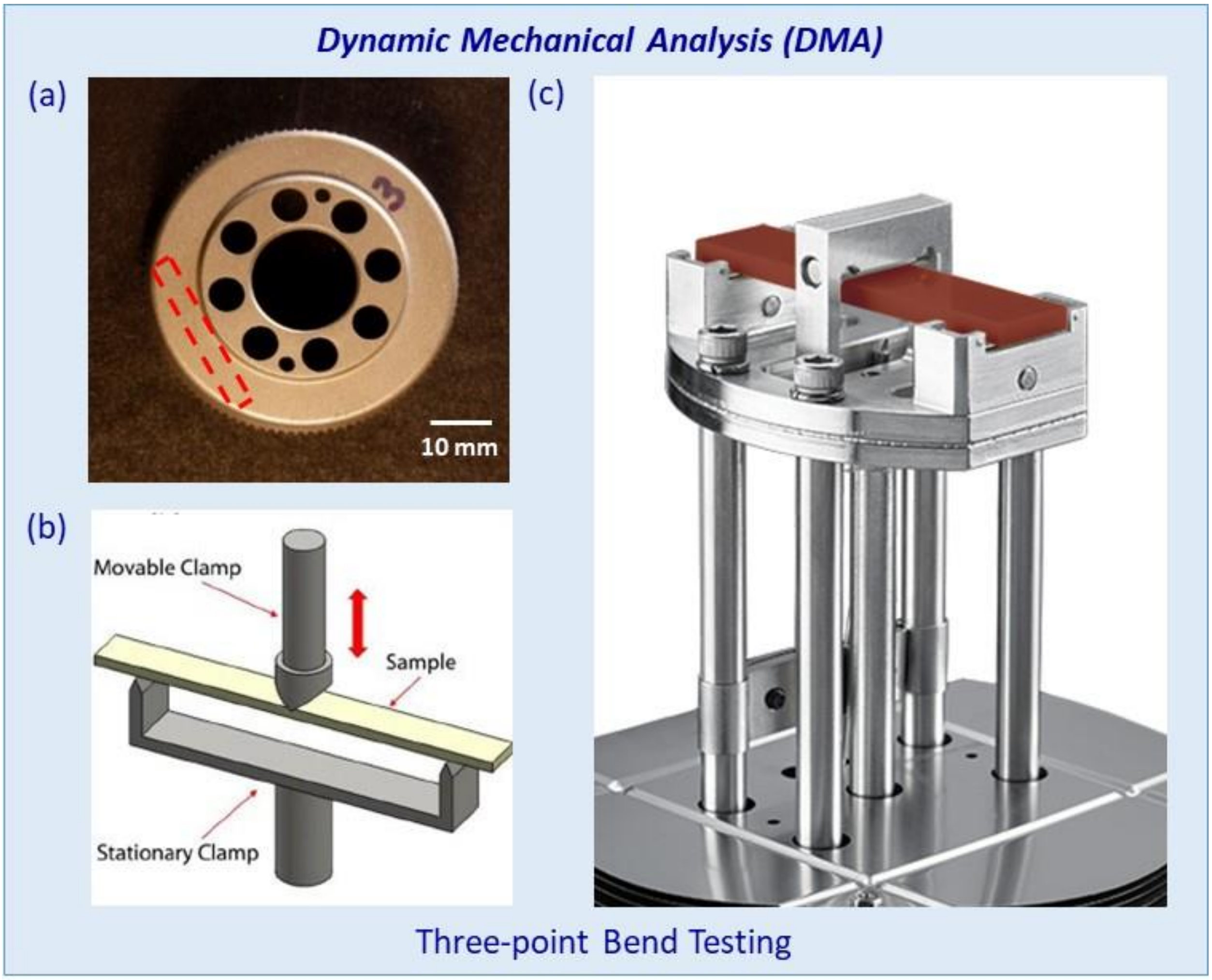


Figure 29. Dynamic mechanical analysis of flat sections extracted from flexspline gears: (a), exemplar of specimen location; (b), generic geometry for static 3-point bend test; (c); typical test fixturing - TA Instruments Q800 [ref. 65].

4.5.2.2. Whole Components

Determining the density of as-cast components is not considered to be a QA test, but is vital to weight-sensitive, structural design. Most techniques have limited resolution for detecting micro-porosity, from the perspective of evaluating casting defects. Measuring the density of components is significant from the perspective of using specific (density-compensated) mechanical properties for structural design. Formal testing procedures may be selected from, but not restricted to

> ASTM B311; Standard Test Method for <u>Density</u> of Powder Metallurgy (PM) Materials Containing Less Than Two Percent Porosity [ref. 41];
>
> ASTM B962; Standard Test Methods for <u>Density</u> of Compacted or Sintered Powder Metallurgy (PM) Products Using Archimedes' Principle [ref. 41]; and

ASTM D2320; Standard Test Method for Density (Relative Density) of Solid Pitch (Pycnometer Method) [ref. 66].

Quantification of casting defects needs to focus on the size and distribution, rather than the volume fraction. For example, some porosity may be permissible in the hub of a gear, but there will likely be zero tolerance in the teeth. Therefore, one of the most important aspects of formal testing of whole components is NDE of inclusions and porosity as a function of location. The penetration depth requirements for flexspline gears, with a wall thickness of ≤ 2mm, facilitate analyses. Techniques that allow multiple components to be evaluated simultaneously would also be beneficial. Chief among the candidate methods is computed tomography, and standard procedures are available: ASTM E1441; Standard Guide for Computed Tomography (CT) Imaging [ref. 59]

ASTM E1570; Standard Practice for Computed Tomographic (CT) Examination [ref. 59]

ASTM E1814; Standard Practice for Computed Tomographic (CT) Examination of Castings [ref. 59]

5. Recommendations

5.1. Adopting a Philosophy

It is evident that a composition-based methodology cannot be employed for QA testing of BMG gear materials until the materials supply chain matures. Therefore, processability- and performance-based methods offer an effective way to verify quality during the manufacture of BMG gears. The risk associated with acceptance of developmental components is at its highest when both a new material and a new processing route are involved. The current issues surrounding flight certification of additively-manufactured (AM) structures represents a comparable situation [refs. 67 and 68]. However, a review of documents relating to qualification of AM parts provided little guidance on formulating a viable protocol in this case. This prompted the decision that the use of an amorphous alloy should be considered secondary, and the fact that the final product is a metal casting should be the priority. The philosophy adopted here is based on tried-and-tested practices established in the ferrous castings industry to minimize the risk. Fortuitously, the approach for creating an effective materials specifications document is outlined in the ***Steel Founders' Society of America*** handbook [ref. 69]:

- ➢ *Testing is required to ensure that castings will perform safely and economically in service*
- ➢ *Excessive testing and overly stringent requirements increase the cost of the product without increasing value*
- ➢ *Insufficient testing or overly lax requirements are meaningless*
- ➢ *It becomes the task of the customer to decide what tests and requirements are necessary for a specific application*

5.2. Division of Responsibilities for QA Testing

The production of witness materials is particularly important during the manufacturing development phase of BMG gears. The approach requires the design and fabrication of separate reference dies dedicated to processability and performance testing. This tooling will be employed to produce acceptance castings and qualification castings. It is essential to employ casting practices for witness materials identical to those used for gear components. Consequently, a synopsis of the division of labor between the parties involved in gear manufacture follows:

- ***Visser*** is responsible for production of "acceptance" castings and "qualification" castings for assessing processability and performance
- ***Materion*** is responsible for screen testing of witness materials for processability
- ***Visser*** is responsible for screen testing of witness materials for performance
- The ***Customer*** is responsible for formal testing of witness materials for processability and performance

5.2.1. Roadmap for Witness Materials

The roadmap for QA testing of witness materials is presented in figure 30. A selection of specimen configurations produced via dedicated castings will be divided between ***JPL and***

LaRC for generating intrinsic material properties. These include physical properties and mechanical properties relevant to the processability and performance of BMG gears for cryogenic applications.

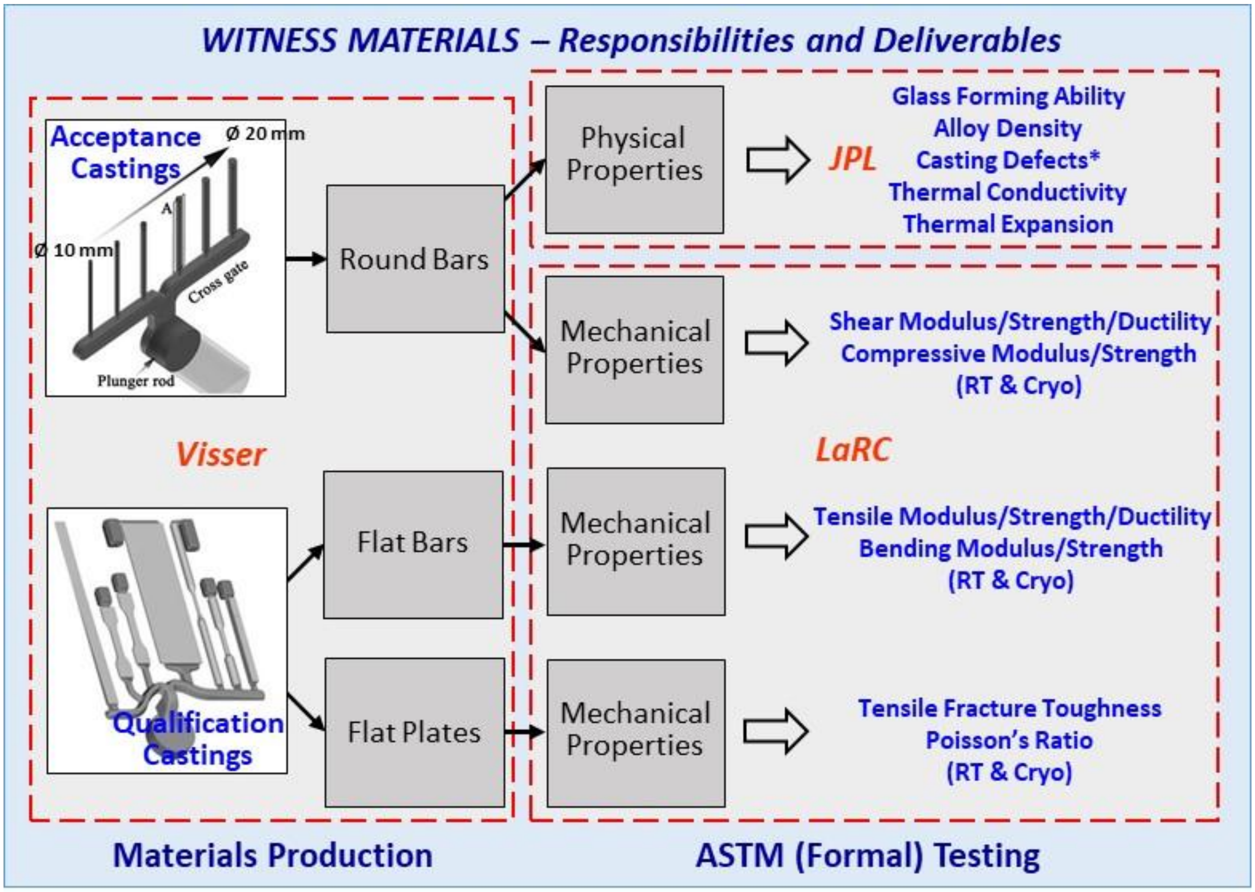


Figure 30. Proposed roadmap for production and QA testing of witness materials from acceptance and qualification castings. A suite of physical and mechanical properties from formal testing is listed. Responsibilities and deliverables of the parties involved are outlined.

5.2.2. Roadmap for Ingots and Castings

Similarly, the roadmap for QA testing of ingots and castings is presented in figure 31. Crystalline re-melt stock will be produced and physical properties screened by ***Materion***. Amorphous gear castings will be produced and physical/mechanical properties screened by ***Visser***. Further screen testing of mechanical properties will be performed at ***LaRC***.

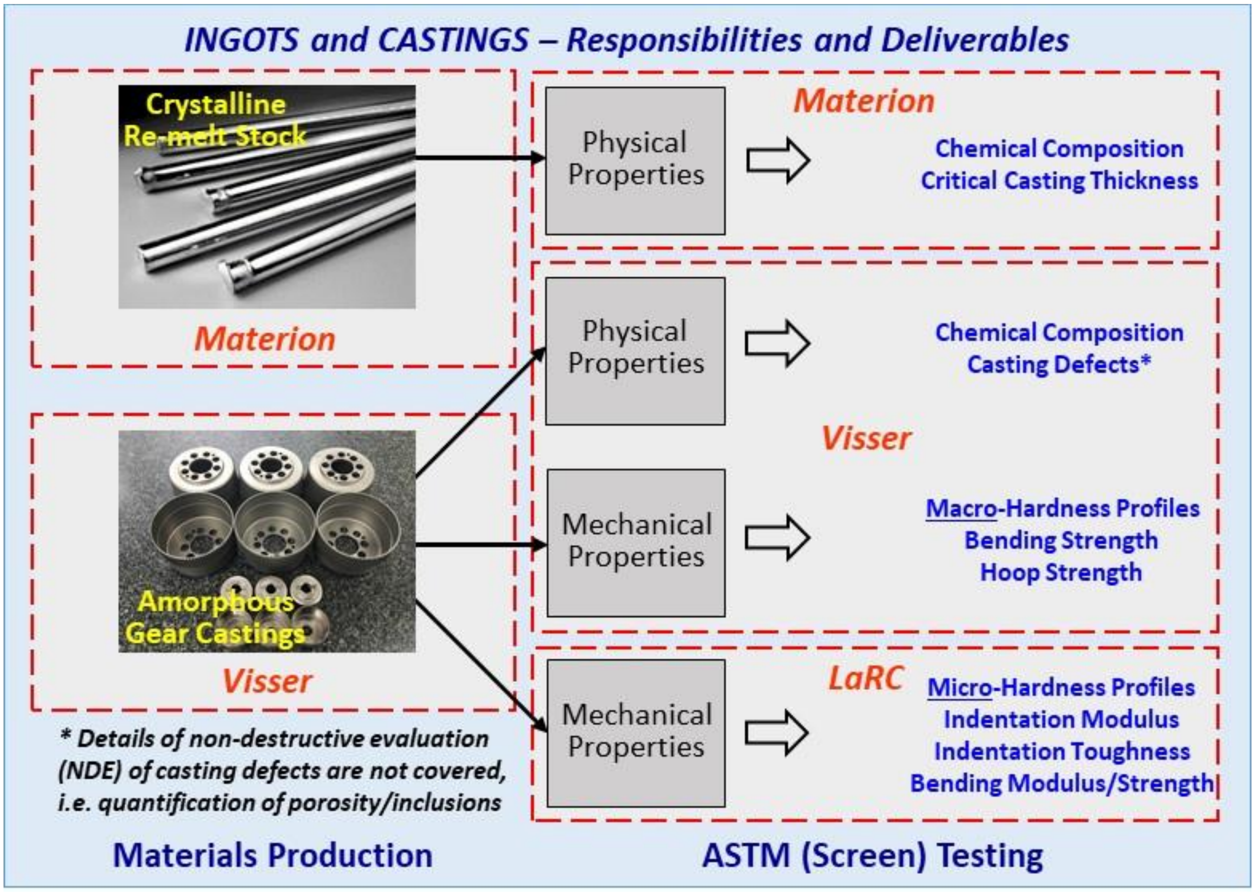
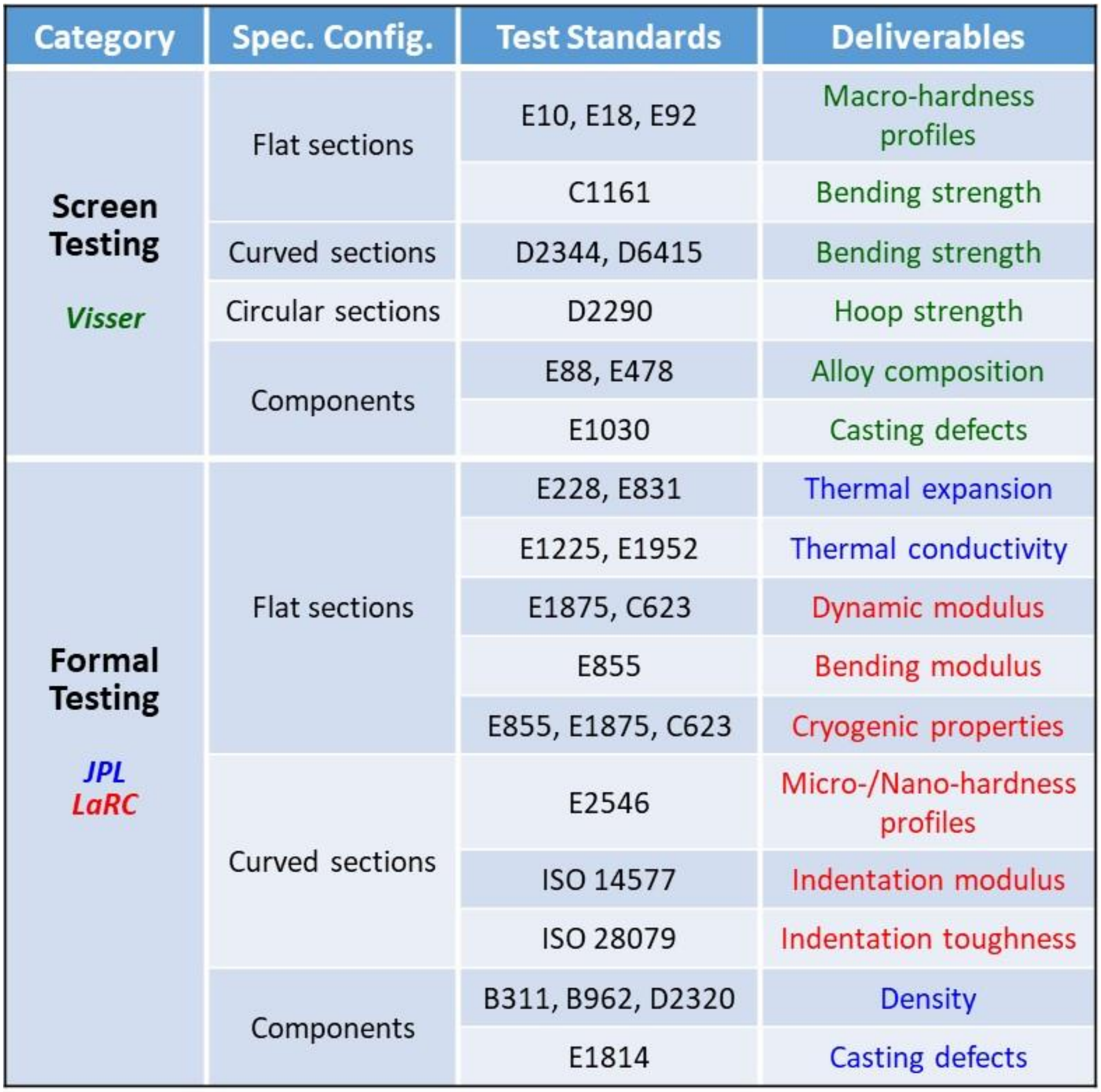


Figure 31. Proposed roadmap for production and QA testing of crystalline ingots and amorphous castings. A suite of physical and mechanical properties from screen testing is listed. Responsibilities and deliverables of the parties involved are outlined.

5.2.3. Specifics for Flexspline Gears

A list of QA tests that are specific to the evaluation of BMG flexspline gears is presented in table 2. It should be noted that any simulated-service testing of gear assemblies at ***JPL*** and ***KSC*** is omitted from these deliberations. The number of intrinsic property tests that can be performed is limited as a consequence of the typical size and shape of fabricated parts. Responsibilities are divided between the simple, screen testing be performed by ***Visser***, and the more-involved, formal testing conducted by ***JPL*** and ***LaRC***. It is suggested that all testing of sectioned or whole components conforms with the ASTM or ISO procedures. The deliverables listed comprise a combination of physical and mechanical properties that define the behavior of the amorphous castings. Substitution of non-standard tests by ***Visser*** is acceptable providing that a detailed rationale with supporting data is furnished.

Table 2. A potential scenario for QA testing of flexspline gears. Responsibilities are divided between screen and formal testing functions. All tests conform with ASTM/ISO procedures. Deliverables comprise a judicious selection of physical and mechanical properties.



Category	Spec. Config.	Test Standards	Deliverables
Screen Testing ***Visser***	Flat sections	E10, E18, E92	Macro-hardness profiles
		C1161	Bending strength
	Curved sections	D2344, D6415	Bending strength
	Circular sections	D2290	Hoop strength
	Components	E88, E478	Alloy composition
		E1030	Casting defects
Formal Testing ***JPL*** ***LaRC***	Flat sections	E228, E831	Thermal expansion
		E1225, E1952	Thermal conductivity
		E1875, C623	Dynamic modulus
		E855	Bending modulus
		E855, E1875, C623	Cryogenic properties
	Curved sections	E2546	Micro-/Nano-hardness profiles
		ISO 14577	Indentation modulus
		ISO 28079	Indentation toughness
	Components	B311, B962, D2320	Density
		E1814	Casting defects

6. Concluding Remarks

This technical paper:

a) provides recommendations to advance materials specifications document JPL D-56223, Rev. A from “draft” to “preliminary” status;
b) proposes division of the current document into two supplier-specific documents;
c) separates testing methods by “ingots/castings” and “witness materials”;
d) classifies test methodology based on “processability” and “performance”;
e) assembles candidate ASTM specifications for “screen testing” and “formal testing”;
f) identifies standard tests compatible with evaluation of BMG flexspline gears;
g) suggests division of responsibilities for processability and performance testing; and
h) addresses the QA “gray area” between production of ingots and castings.

The report recommends that:

a) processability- and performance-based QA methods might be used *in lieu* of compositional specifications during manufacturing development only;
b) reference dies be designed for “acceptance” and “qualification” castings;
c) witness materials provide physical and mechanical property data;
d) formal testing be conducted at ambient and cryogenic temperatures; and
e) processing/properties be correlated with compositional variations.

7. Selected Resources

7.1. Professional Organizations

American Gear Manufacturers Association (AGMA), Alexandria, VA; https://www.agma.org/

American National Standards Institute (ANSI), Washington, DC; https://www.ansi.org/

American Society for Testing and Materials (ASTM), West Conshohocken, PA; https://www.astm.org/

International Organization for Standardization (ISO), Geneva, Switzerland; https://www.iso.org/

North American Die Casting Association (NADCA), Arlington Heights, IL; https://www.diecasting.org/

Society of Automotive Engineers (SAE), Warrendale, PA; https://www.sae.org/

Steel Founders' Society of America (SFSA), Crystal Lake, IL; https://www.sfsa.org/

7.2. Government Agencies

Metallic Materials Properties Development and Standardization (MMPDS), FAA/Battelle, Columbus, OH; https://www.mmpds.org/

NASA Technical Standards System (NTSS), Washington, DC; https://standards.nasa.gov/

National Institute of Standards and Technology (NIST), Gathersburg, MD; https://www.nist.gov/

United States Patent and Trademark Office (USPTO), Washington, DC; https://www.uspto.gov/

7.3. Relevant US Patents

7.3.1. Alloy S77PT Composition

Y.C. Kim, E. Fleury, K.B. Kim, and H.K. Seok, "Cu-based amorphous alloy composition," US Patent # 7,147,727 B2, September 12, 2006.

7.3.2. Casting of BMGs

D.C. Hofmann and A. Kennett, "Systems and methods for fabricating structures including metallic glass-based materials using low pressure casting," US Patent # 9,868,150 B2, January 16, 2018.

H. Fu ..... and T. Tang, "Bulk amorphous alloy Zr-Cu-Ni-Al-Ag-Y and methods of preparing and using the same," US Patent # 9,896,753 B2, February 20, 2018.

W.L. Johnson, C. Kim, and A. Peker, "Thermoplastic casting of amorphous alloys," US Patent # 7,017,645 B2, March 28, 2006.

C.D. Prest, M.S. Scott, D.J. Stratton, J.C. Poole, and T.A. Waniuk, “Method for quantifying amorphous content in bulk metallic glass parts using thermal emissivity,” US Patent # 8,829,437 B2, September 9, 2014.

T.A. Waniuk ..... and C.D. Prest, “Injection compression molding of amorphous alloys,” US Patent # 9,649,685 B2, May 16, 2017.

7.3.3. SWG Fabrication

D.C. Hofmann and B. Wilcox, “Method for manufacturing bulk metallic glass-based strain wave gear components,” US Patent # 9,791,032 B2, October 17, 2017.

D.C. Hofmann and B. Wilcox, "Systems and methods for implementing bulk metallic glass-based strain wave gears and strain wave gear components," US Patent # 9,328,813 B2, May 3, 2016.

8. References

1. D.C. Hofmann ..... and A. Kennett, “Optimizing bulk metallic glasses for robust, highly wear-resistant gears,” Adv. Eng. Mater., vol.19, no. 1, paper# 1600541, 10 pp., 2017.
2. D.C. Hofmann ..... and B. Wilcox, “Castable bulk metallic glass strain wave gears: Towards decreasing the cost of high-performance robotics,” Scientific Reports, vol. 6, paper# 37773, 11 pp., 2016.
3. D.C. Hofmann and A. Kennett, “Low-pressure casting of bulk metallic glasses for gears and other applications,” Tech Briefs, vol. 39, no. 7, 2015.
4. J.R. Davis (ed.), ***Gear materials, properties, and manufacture***, ASM International, Materials Park, OH, 329 pp., 2005.
5. D.C. Hofmann, A. Kennett, and K.T. Boykins, “Developing ceramic-like bulk metallic glass gears,” Tech Briefs, vol. 39, no. 3, 2015.
6. L.M. Andersen, D.C. Hofmann, and K.S. Vecchio, “Effect of zirconium purity on the glass-forming-ability and notch toughness of $Cu_{43}Zr_{43}Al_7Be_7$,” Mater. Sci. Eng, vol. A674, pp. 397-405, 2016.
7. ***Materion***, Elmore, OH; https://materion.com/businesses/beryllium-and-composites
8. ***Visser Precision Cast***, Denver, CO; https://visserprecision.com/
9. R.P. Dillon, “Bulk metallic glass gears,” JPL, Pasadena, CA; Presentation at NASA Langley Research Center, Hampton, VA, March 27, 2017.
10. Y.C. Kim ..... and E. Fleury, “Enhanced glass forming ability and mechanical properties of new Cu-based bulk metallic glasses,” Mater. Sci. & Eng., vol. A437, pp. 248-253, 2006.
11. S.W. Lee, M.Y. Huh, E. Fleury, and J.C. Lee, “Crystallization-induced plasticity of Cu–Zr containing bulk amorphous alloys,” Acta Mater., vol. 54, pp. 349-355, 2006.
12. C. Gonzalez, “Gears look to the future for material,” Machine Design, https://www.machinedesign.com/engineering-essentials/gears-look-future-material, December 2015.
13. S. Ding ..... and J. Schroers, “Combinatorial development of bulk metallic glasses,” Nature Materials, vol. 13, no. 5, pp. 494-500, 2014.
14. R. Busch, “The thermophysical properties of bulk metallic glass-forming liquids,” JOM, vol. 52, no. 7, pp. 39-42, 2000.
15. J. Cheng ..... and R. Schmid-Fetzer, “Measurement and calculation of the viscosity of metals—a review of the current status and developing trends,” Measurement Sci. & Tech., vol. 25, no. 6, 2014.
16. ***Copper and Copper Alloys***, ASTM, Conshohocken, NJ, vol. 02.01, 1046 pp., May 2018.
17. ***Analytical Chemistry for Metals, Ores, and Related Materials***, ASTM, Conshohocken, NJ, vol. 03.05, 1156 pp., October 2018.
18. A.R. Anilchandra ..... and G. Timelli, “Evaluating the tensile properties of aluminum foundry alloys through reference castings—A review,” Materials, vol. 10, no. 9, pp. 1011-1023, 2017.
19. ***An Introduction to Die Casting***, American Die Casting Institute, Inc., New York, NY, 30 pp., 1965.
20. W.L. Johnson, J.H. Na, and M.D. Demetriou, “Quantifying the origin of metallic glass formation,” Nature Communications, vol. 7, paper# 10313, 7 pp., January 2016.
21. K.J. Laws, B. Gun, and M. Ferry, “Influence of casting parameters on the critical casting size of bulk metallic glass,” Metall. & Mater. Trans., vol. 40A, no. 10, pp. 2377-2387, 2009.

22. D.V. Neff, “Improving die casting melt quality and casting results with melt quality analysis and filtration,” NADCA paper, https://www.dykast.com/user/products/5689-pdfs-1-file.pdf, 12 pp., October 2002.
23. ***Liquidmetal Design Guide V4.2***, Liquidmetal Technologies, Rancho Santa Margarita, CA: https://www.liquidmetal.com/wp-content/uploads/2017/11/DesignGuide4.2_v6_Web.pdf, 84 pp., November 2017.
24. S. Ji ..... and Z. Fan, “Effect of iron on the microstructure and mechanical property of Al–Mg–Si–Mn and Al–Mg–Si diecast alloys,” Mater. Sci. & Eng., vol. A564, pp. 130-139, 2013.
25. ***Izod and Charpy Specimens***, NIST, Gathersburg, MD: https://www.nist.gov/mml/acmd/structural-materials-group/izod, September 2018.
26. ***Metals – Mechanical Testing; Elevated and Low-Temperature Tests; Metallography***, ASTM, Conshohocken, NJ, vol. 03.01, 1776 pp., July 2018.
27. ***Fracture Toughness Testing***, Laboratory Testing, Inc., Hatfield, PA: https://www.labtesting.com/services/materials-testing/fracture-mechanics/fracture-toughness/, September 2018.
28. ***Fracture Toughness: Part Two***, Key to Metals North America, St. Louis, MO: https://www.totalmateria.com/page.aspx?ID=CheckArticle&site=kts&LN=EN&NM=294, November 2010.
29. R.A. Hafley, M.S. Domack, S.J. Hales, and R.N. Shenoy, ***Evaluation of Aluminum Alloy 2050-T84 Microstructure and Mechanical Properties at Ambient and Cryogenic Temperatures***, NASA Langley Research Center, Hampton, VA, NASA/TM-2011-217163, 98 pp., July 2011.
30. M. Niedzinski and P. Bittner, “Emergence of Airware® 2050 for space launch cryogenic applications,” Constellium, Ravenswood, WV; Presentation at NASA Langley Research Center, Hampton, VA, August 2, 2018.
31. Y. Zhou ..... and Q. Zhai, “Reduced fracture toughness of metallic glass at cryogenic temperature,” Metals, vol. 7, no. 4, paper# 151, 11 pp., 2017.
32. S. Roberts ..... and D.C. Hofmann, “Cryogenic Charpy impact testing of metallic glass matrix composites,” Scripta Mater., vol. 66, no. 5, pp. 284-287, 2012.
33. G. Li ..... and J. Sun, “The ductile to brittle transition behavior in a Zr-based bulk metallic glass,” Mater. Sci. & Eng., vol. A625, pp. 393-402, 2015.
34. L.H. Liu ..... and Z.Y. Liu, “Determination of forming ability of high pressure die casting for Zr-based metallic glass,” Mater. Proc. Techol., vol. 244, pp. 87-96, 2017.
35. F. Ren ..... and A. Mehta, “Accelerated discovery of metallic glasses through iteration of machine learning and high-throughput experiments,” Science Advances, vol. 4, ID 1566, 11 pp., 2018.
36. D. Schwam, J.F. Wallace, Q. Chang, and Y. Zhu, “Optimization of the squeeze casting process for aluminum alloy parts,” Case Western Reserve University, Dept. of Energy Report # 77DE-FC07-98ID13613, 156 pp., July 2002.
37. D. Caliari ..... and P. Giordano, “Fluidity of aluminium foundry alloys: Development of a testing procedure,” La Metallurgia Italiana, vol. 107, no. 6, pp. 11-18, 2015.
38. R. Busch, J. Schroers, and W.H. Wang, “Thermodynamics and kinetics of bulk metallic glass,” MRS Bulletin, vol. 32, no. 8, pp. 620-623, 2007.
39. Y.J. Kim ..... and W.K. Rhim, “Experimental determination of a time–temperature-transformation diagram of the undercooled $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10.0}Be_{22.5}$ alloy using the

containerless electrostatic levitation processing technique," Appl. Phys. Lett., vol. 68, no. 8, pp. 1057-1059, 1996.

40. ***Statistical Methods; Hazard Potential of Chemicals; Thermal Measurements; Manufacture of Pharmaceutical and Biopharmaceutical Products***, ASTM, Conshohocken, NJ, vol. 14.05, 1338 pp., July 2018.
41. ***Metallic and Inorganic Coatings; Metal Powders and Metal Powder Products***, ASTM, Conshohocken, NJ, vol. 02.05, 1104 pp., May 2018.
42. G. Singh and A. Verma, "A brief review on injection moulding manufacturing process," Materials Today, vol. 2, no. 4, pp. 1423-1433, 2015.
43. ***Aluminum and Magnesium Alloys***, ASTM, Conshohocken, NJ, vol. 02.02, 910 pp., September 2018.
44. ***Plastics (II): D3222–D5083***, ASTM, Conshohocken, NJ, vol. 08.02, 916 pp., June 2018.
45. ISO 294-2, "Plastics — Injection moulding of test specimens of thermoplastic materials," Int. Org. Standards, Geneva, Switzerland, 6 pp., December 1996.
46. ISO 20753, "Plastics — Test specimens," Int. Org. Standards, Geneva, Switzerland, 22 pp., March 2008.
47. ***Refractories, Activated Carbon; Advanced Ceramics***, ASTM, Conshohocken, NJ, vol. 15.01, 1266 pp., March 2018.
48. ISO 6892-3, "Metallic materials — Tensile testing," Int. Org. Standards, Geneva, Switzerland, 30 pp., April 2015.
49. ISO 15579, "Metallic materials — Tensile testing at low temperature," Int. Org. Standards, Geneva, Switzerland, 22 pp., June 2000.
50. C.A. Schuh, T.C. Hufnagel, and U. Ramamurty, "Mechanical behavior of amorphous alloys," Acta Materialia, vol. 55, no. 12, pp. 4067–4109, 2007.
51. J. Yi, S.M. Seifi, W. Wang, and J.J. Lewandowski, "A damage-tolerant bulk metallic glass at liquid-nitrogen temperature," Mater. Sci. Technol., vol. 30, no. 6, pp. 627-630, 2014.
52. H. Chandler (ed.), ***Hardness Testing***, 2nd Edition, ASM International, Materials Park, OH, 192 pp., 1999.
53. ISO 14577-1, "Metallic materials — Instrumented indentation test for hardness and materials parameters," Int. Org. Standards, Geneva, Switzerland, 54 pp., July 2015.
54. "Palmqvist method," ***Wikipedia***, *The Free Encyclopedia*, last modified March 10, 2018, https://en.wikipedia.org/wiki/Palmqvist_method. Accessed October 26, 2018. [Created by RefDr; image licensed under https://creativecommons.org/licenses/by-sa/4.0/].
55. ISO 28079, "Hardmetals – Palmqvist toughness test," Int. Org. Standards, Geneva, Switzerland, 18 pp., July 2009.
56. G.D. Quinn and R.C. Bradt, "On the Vickers indentation fracture toughness test," Am. Ceram. Soc., vol. 90, no. 3, pp. 673-680, 2007.
57. ***Space Simulation; Aerospace and Aircraft; Composite Materials***, ASTM, Conshohocken, NJ, vol. 15.03, 1594 pp., October 2018.
58. ***Plastic Piping Systems***, ASTM, Conshohocken, NJ, vol. 08.04, 1848 pp., January 2018.
59. ***Nondestructive Testing (I): C1331 – E2373***, ASTM, Conshohocken, NJ, vol. 3.03, 1402 pp., October 2018.
60. K.P. Menard, ***Dynamic Mechanical Analysis – A practical introduction***, 2nd Edition, CRC Press, Boca Raton, FL, 218 pp., 2008.

61. J.M. Pelletier and B. Van de Moortele, “Mechanical properties of bulk metallic glasses: Elastic, visco-elastic, and visco-plastic components in the deformation,” Non-Crystalline Solids, vol. 353, pp. 3750-3753, 2007.
62. ***Glass; Ceramic Whitewares***, ASTM, Conshohocken, NJ, vol. 15.02, 632 pp., April 2018.
63. ***Plastics (I): C1147–D3159***; ASTM, Conshohocken, NJ, vol. 08.01, 942 pp., June 2018.
64. ISO 178, “Plastics — Determination of flexural properties,” Int. Org. Standards, Geneva, Switzerland, 24 pp., December 2010.
65. ***DMA Q800, Thermal Analysis***, TA Instruments, Inc., New Castle, DE: http://www.tainstruments.com/wp-content/uploads/dma.pdf, 22 pp., January 2011.
66. ***Petroleum Products, Liquid Fuels, and Lubricants (I): C1234–D3709***, ASTM, Conshohocken, NJ, vol. 5.01, 1844 pp., February 2018.
67. ***Standard for Additively Manufactured Spaceflight Hardware by Laser Powder Bed Fusion in Metals***, NASA Marshall Spaceflight Center, Huntsville, AL, MSFC-STD-3716, 93 pp., October 2017.
68. ***Specification for Control and Qualification of Laser Powder Bed Fusion Metallurgical Processes***, NASA Marshall Spaceflight Center, Huntsville, AL, MSFC-STD-3717, 58 pp., October 2017.
69. “Summary of standard specifications for steel castings,” ***Steel Castings Handbook***, Steel Founders’ Society of America, Crystal Lake, IL, Supplement 2, 66 pp., 2009.

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14. ABSTRACT

NASA is evaluating bulk metallic glass (BMG) gears for extreme environment (cryogenic) applications; e.g., Europa Lander. The main purpose of this report is to recommend a quality assurance (QA) protocol for the production of consistent and reliable gear castings. Currently, there are two, separate manufacturers involved; Materion Corporation produces re-melt stock and Visser Precision Cast, Inc. produces BMG castings. Division of the existing alloy specifications document into material supplier-specific documents is proposed. Until a composition-based specification is established, processability- and performance-based methodologies may be an option during manufacturing development. It is recommended that each of the documents has a distinct focus; the processing behavior of crystalline feedstock, and the mechanical behavior of amorphous castings.

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