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**Dynamic Devitrification of Bulk Metallic Glasses (BMGs)
and Composites of BMG With Tungsten**

by Laszlo J. Kecskes, Robert H. Woodman, and Samuel F. Trevino

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14. ABSTRACT The penetration behavior of tungsten-bulk metallic glass (BMG) matrix composites is expected to be strongly influenced by the deformation behavior of the BMG matrix. It has been hypothesized that when the BMC matrix, initially in its viscous, deeply undercooled state, is subjected to compressive loads under the rapid time scales during ballistic impact, it may become a liquid without devitrification. We have carried out a series of in-situ x-ray diffraction experiments at the Advanced Photon Source (APS) and the National Synchrotron Light Source (NSLS) on BMG samples while heating them. At the same time, we carried out differential thermal analysis experiments and analyzed the results using the Kissinger method to estimate the critical heating rates necessary to bypass devitrification. The data collected and results show that the heating rates available at either APS or NSLS were insufficient to verify the hypothesis.					
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1. Objective

The objective of this investigation is to demonstrate that bulk metallic glasses (BMG) can be melted without undergoing devitrification. This would establish a basis for testing whether or not BMG-matrix penetrators undergo matrix melting during penetration.

Microscopic examination of a rod of bulk metallic glass fired into a semi-infinite, rolled-homogeneous-armor (RHA) target yields evidence that the glass melts during penetration (figure 1). Residue from the rod on the penetrator wall exhibited cracks normally associated with solidification from a melt (known as shrinkage cracks). This, along with similar evidence from residual composite penetrators, led to the hypothesis that molten matrix material is ejected from tungsten-BMG composite penetrators during penetration.

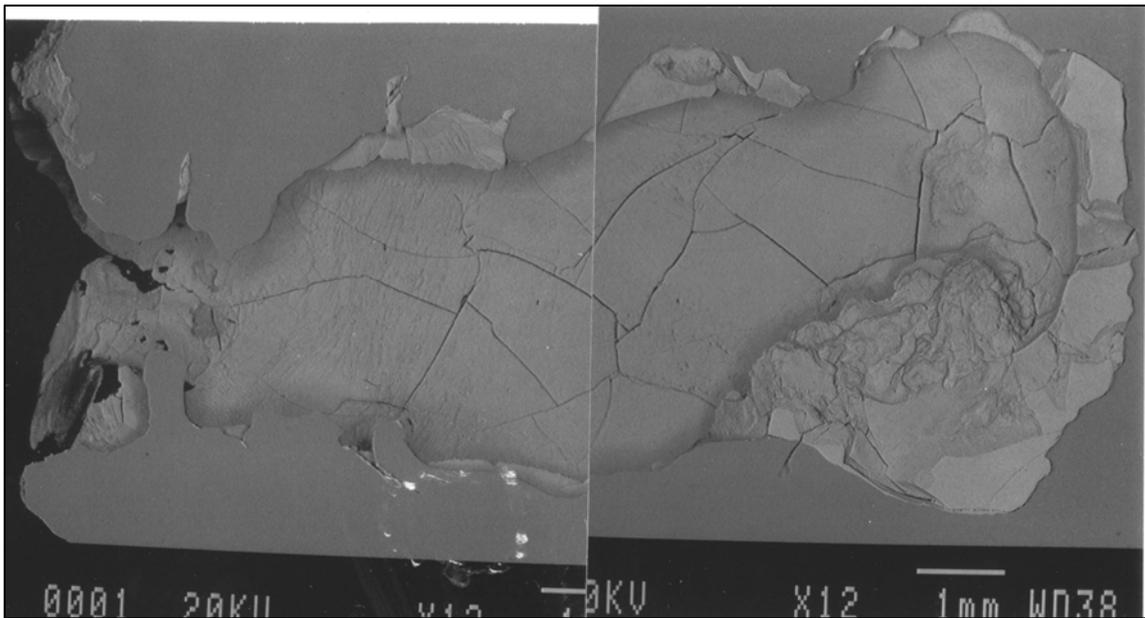


Figure 1. Residual, glass-only BMG penetrator in RHA target. Entry point is to the left.

If the matrix does melt during penetration, interpretation of the limit-velocity tests used to evaluate the effectiveness of penetrators will change. The limit velocity of BMG-tungsten composites has been interpreted as being due to a combination of the characteristic erosion mechanisms observed in two other materials—depleted uranium (DU) and tungsten heavy alloy (WHA).

All other factors being equal, a more dense penetrator will exhibit a lower limit velocity, i.e., its penetration performance will be superior to a less dense material. WHA, which can be prepared in a range of densities from 16 to 19 g/cm³, exhibits this trend (figure 2). However, while DU alloys follow a similar trend, they also exhibit limit velocities measurably below those of WHAs

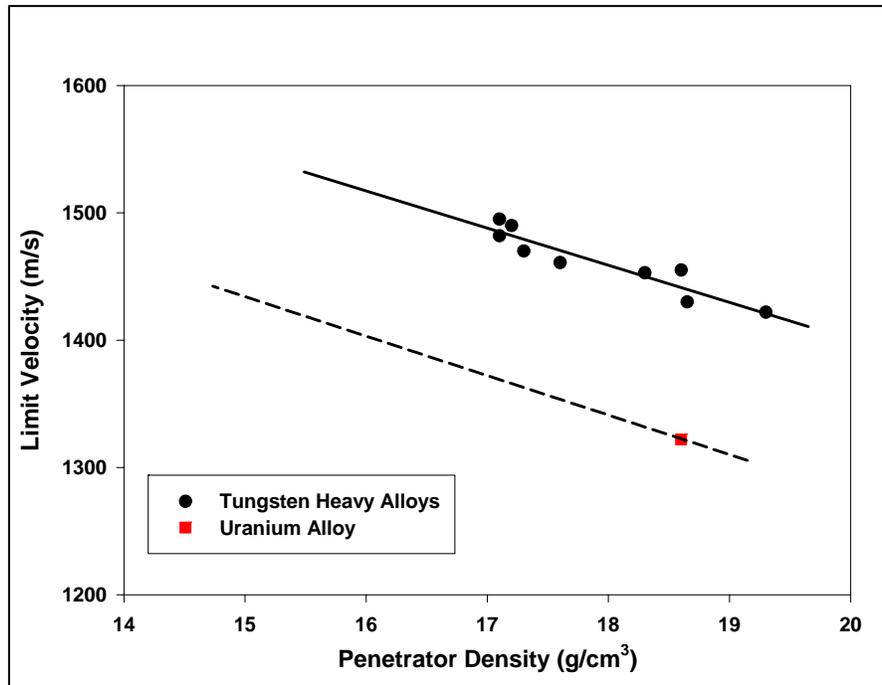


Figure 2. Plot of limit velocity vs. density for various alloys.

of comparable density. Furthermore, while DU alloys are less dense than pure tungsten, they exhibit superior penetration performance.

It has been established that the penetration performance of DU relative to WHA is due to a difference in the erosion-and-discard mechanism for material at the penetrator tip (*I*). In tungsten and WHA, plastic deformation results in a broadening of the penetrator nose, which leads to a wider penetration tunnel than the initial diameter of the rod. In contrast, localized deformation of DU leads to sharpening of the nose, which leads to an overall greater depth of penetration.

Tungsten-BMG composites were developed because metallic glasses deform and fracture by a localized mechanism similar to that seen in DU. Accordingly, the penetration performance of tungsten-BMG composites, which is intermediate between that of WHA and DU, has been interpreted as being a mix of plastic deformation and shear localization.

If, in fact, molten metal is ejected from a tungsten-BMG composite during penetration, the limit velocities of such composites would need to be appreciated in light of such a third mechanism. The composite performance would be expected to reflect a mixture of the effect of ejection of molten matrix and plastic deformation of the tungsten. The nature of the limit-velocity-vs.-density relationship for material which melts during penetration has not yet been studied.

Much of the kinetic energy dissipated during penetration results in heating of both penetrator and target. What is unknown is whether or not the temperature rises high enough to exceed the glass-transition temperature (~500 °C) or liquidus temperature (~1000 °C) of a BMG. If the glass-

transition temperature is exceeded, and the glass does not devitrify, the pressures experienced during penetration might be sufficient to cause the glass to behave like molten material. If the liquidus is exceeded, regardless of devitrification the matrix will be molten.

Ballistic test facilities are not yet equipped to monitor temperature and microstructural changes in either penetrator or target (it would also be a challenge to adapt existing technology to do so in situ). Furthermore, because BMGs are a recent development, there are not yet physical models that might be used to estimate localized temperatures during penetration.

To demonstrate that melting during penetration is possible, we undertook to monitor the structural changes in BMGs while heating them as rapidly as possible with available equipment.

2. Approach

In order to show that BMGs can be melted without devitrifying, we collected x-ray diffraction patterns from samples while heating them. The diffraction pattern of the amorphous structure of the glass differs noticeably from that of the crystalline devitrified material. If a sample were melted without devitrifying, it would retain the characteristic pattern of glasses and liquids throughout heating. If, on the other hand, the glass devitrified during heating, crystalline patterns would appear during the scan. In order to collect diffraction data of sufficient resolution, we found it necessary to use x-rays generated at synchrotron sources—the Advanced Photon Source (APS) at Argonne National Laboratory, and the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory.

For the same reasons that cooling a melt to a glass without crystallization requires rapid cooling, melting a glass without devitrification requires rapid heating. Making glasses requires a minimum cooling rate—the critical cooling rate—to be achieved (2). Melting without devitrification requires that a critical heating rate be achieved. In order to estimate the critical heating rate, we conducted differential thermal analysis (DTA) of BMG samples and analyzed the results using the Kissinger method (3–6). Application of the Kissinger method to the devitrification kinetics of glasses has been analyzed in detail by Henderson (7).

The Kissinger method relates the variation of DTA peak temperatures with heating rate to the activation energy of the physical process associated with the peak. A similar analysis can be used to calculate the heating rate which would be required to shift the peak temperature above a specific value. If the liquidus temperature is selected as the specific value, then the calculated heating rate is an estimate of the critical heating rate.

2.1 Materials

Two different BMGs were used in this study. Table 1 lists their names, compositions, glass-transition temperature (T_g), liquidus temperature (T_L), and density. Vitreloy 105 is one of many zirconium-based BMG-forming alloys developed at the California Institute of Technology in the 1990s (8). This class of alloys, known as Vitreloys, included some of the first BMGs to be made in sizes larger than 10 mm. Hf 105 is similar in composition to Vitreloy 105, with the zirconium replaced fully by hafnium, and was developed under the U.S. Army Research Laboratory's Materials Center of Excellence program with Johns Hopkins University (9). Hf 105 represents an attempt to improve the ballistic performance of BMG-tungsten composites by raising the density of the BMG. Substitution of Hf for Zr in this formula increases the alloy density, but has deleterious effects on the glass-forming ability of the alloy.

Table 1. BMG test results.

Name	Composition	T_g (°C)	T_L (°C)	Density (g/cm ³)
Vitreloy 105	Zr _{52.5} Ti ₅ Cu _{17.9} Ni _{14.6} Al ₁₀	400	862	6.7
Hf 105	Hf _{52.5} Ti ₅ Cu _{17.9} Ni _{14.6} Al ₁₀	467	1000	11.1

2.2 In-situ X-ray Diffraction

Tests at the APS were conducted at experimental station 6-ID-D (10). This instrument, administered by the Midwestern Universities Collaborative Access Team, delivers and detects x-rays with energies of 49.5–132.3 keV. This is 6–16 times higher than the 8.04 keV x-rays generated by typical laboratory x-ray generators. Consequently, the x-rays can fully penetrate samples which are 2–3 mm thick. Accordingly, the diffraction geometry for the experiments was in transmission, similar to electron diffraction in a transmission electron microscope.

Diffraction patterns were collected using a charge-coupled device (CCD) camera detector (11). The camera was exposed to the scattered x-rays from the samples for 5-s spans at regular intervals during heating. A sample of suction-cast Vitreloy 105 was heated in an electric furnace in the incident x-ray beam at 6 °C/min from 350 to 500 °C. A total of 176 5-s exposures of the CCD camera were recorded during heating. Raw images from the CCD camera were analyzed using FIT2D, a program developed at the European Synchrotron Research Facility in Grenoble, France (12).

Tests at the NSLS were conducted on beamline X-7B (13). The wavelength of the x-rays was 0.92292 Å. A two-dimensional, position-sensitive detector was used to record the diffraction pattern. Samples of melt-spun Vitreloy 105 and Hf 105 were mounted in a tube of single-crystal sapphire heated by a high-resistance coil. A thermocouple inserted in the tube was used for temperature measurement. Diffraction patterns were recorded from samples of Vitreloy 105 heated at 1.2, 2.4, and 6.4 °C/min, and from samples of Hf 105 heated at 1.67, 4.44, and 11.92 °C/min.

2.3 Estimate of Critical Heating Rate

Differential thermal analysis was performed using a Netzsch Instruments Simultaneous Thermal Analyzer model 409C, using graphite crucibles in an argon atmosphere. Samples of suction-cast Hf 105 were heated at four heating rates (5, 10, 20, and 40 K/min).

Calculation of the critical heating rate involves rearranging the Kissinger-method equation:

$$\frac{d\left[\ln\left(\beta/[T-T_0]^2\right)\right]}{d(1/[T-T_0])} = -\frac{E}{R}, \quad (1)$$

where β is the heating rate, T is the peak temperature, E is the activation energy, and R is the gas-law constant. T_0 is a constant temperature determined from a fit of the DTA data to the Vogel-Tammann-Fulcher equation (14), following the procedure described by Henderson (7).

It is clear from equation 1 that a plot of $\ln\left(\beta/[T-T_0]^2\right)$ vs. $1/[T-T_0]$ will have a slope of $-E/R$. That is:

$$\ln\left(\frac{\beta}{[T-T_0]^2}\right) = M\left(\frac{1}{[T-T_0]}\right) + B, \quad (2)$$

where M is the slope and B is the intercept of the plot. Substituting the liquidus temperature of the alloy, T_L , for T , β becomes the critical heating rate, β_{crit} . In other words, β_{crit} can be determined directly from the plot:

$$\beta_{crit} = (T_L - T_0)^2 \exp\left[\frac{M}{(T_L - T_0)} + B\right]. \quad (3)$$

3. Results

3.1 In-situ X-ray Diffraction

The first and last of the 176 diffraction patterns recorded at APS are presented in figures 3 and 4. It is quite clear from figure 4 that the sample devitrified during heating. The diffuse ring visible in figure 3, characteristic of scattering from liquids and glasses, has been replaced in figure 4 by the distinct diffraction lines of a polycrystalline material. It is evident from this that 6 °C/min is lower than the critical heating rate.

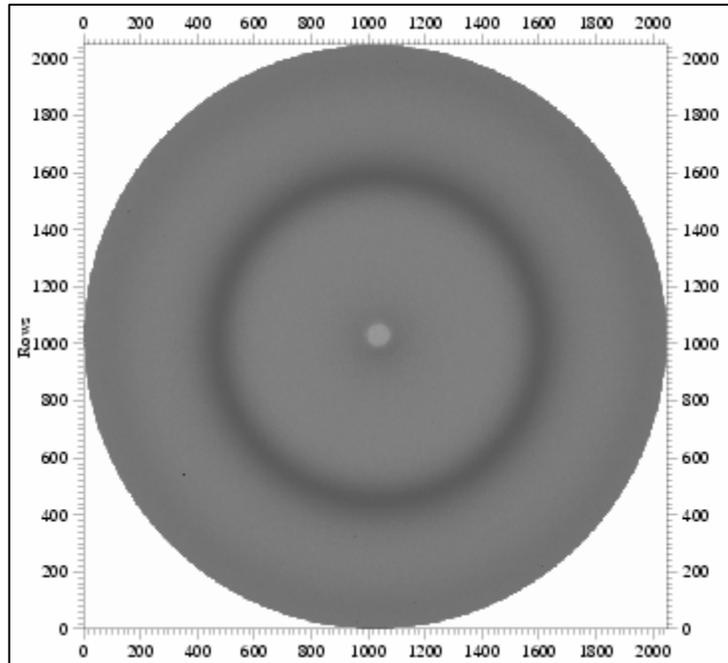


Figure 3. Debye-Scherrer photograph of Vitreloy 105 at room temperature.

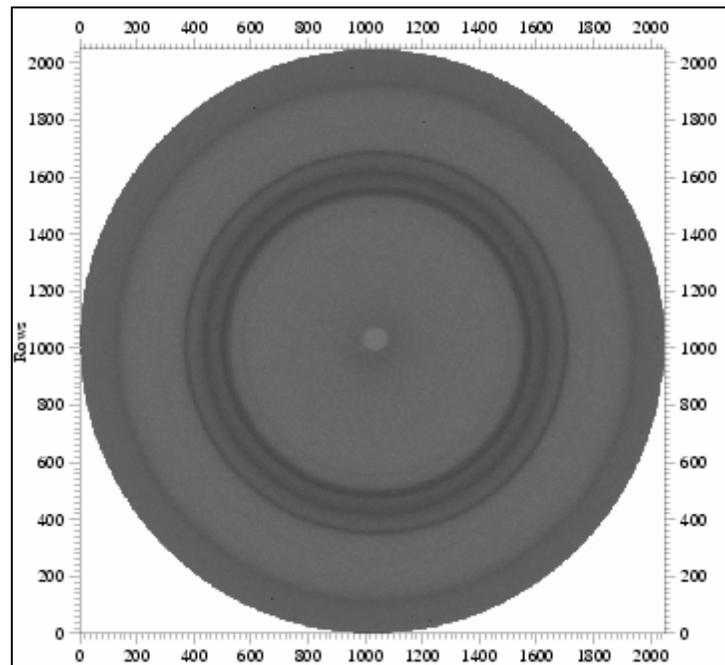


Figure 4. Debye-Scherrer photograph of Vitreloy 105 at 500 °C.

Figure 5 is a false-color image of the diffraction patterns from Vitreloy 105 across the temperature range 25–600 °C at a heating rate of 6.39 °C/min. Color represents scattering intensity as a function of Q ($4\pi\sin(\theta)/\lambda$, where 2θ is the scattering angle and λ the wavelength) and temperature. Figure 5 is a top-down view of the series of diffraction patterns as the

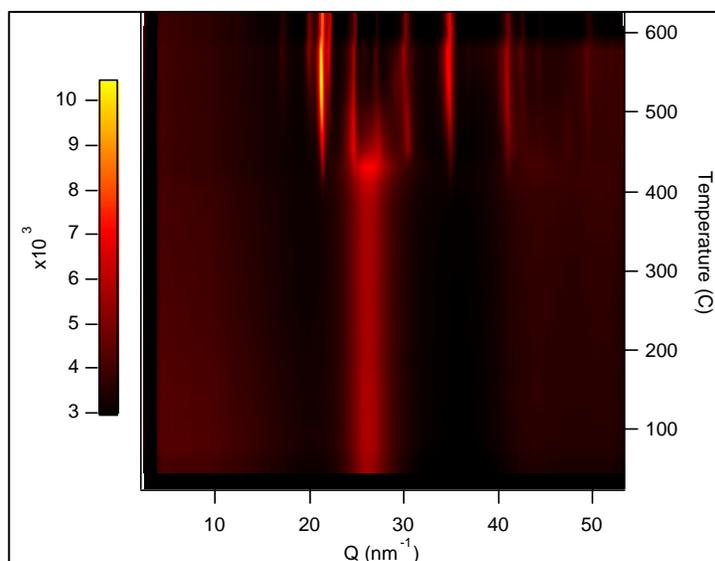


Figure 5. False-color image of XRD patterns from Vitreloy 105 taken at 6.39 °C/min.

temperature rises. The broad streak evident from room temperature up to just over 400 °C is the diffuse scattering from the glass structure. It is analogous to the diffuse ring in figure 3. The more numerous, sharp streaks at higher temperatures are the diffraction peaks from the devitrified material. It is clear from figure 5 that the glass devitrifies between 400 and 500 °C. The diffuse band between 20 and 30 nm⁻¹ is the broad scattering peak from a glass structure, while the bright streaks at higher temperatures are the diffraction peaks from the crystalline structure.

Figure 6 plots the integrated intensity vs. temperature for the peak at $Q = 21.3 \text{ nm}^{-1}$. Calculated averages of raw data in a finite Q interval centered at a given Q value are shown as data points (this is the same as the integrated intensity of a peak centered on a given value of 2θ). The data for each heating rate are scaled to obtain comparable maximum intensities. It is clear from the plot that the effect of increasing the heating rate is to expand the range of temperature over which the devitrification reaction occurs. Plots for the peaks at 34.7 nm^{-1} and 40.9 nm^{-1} have a similar appearance.

Figure 7 is a plot of the temperature, $T_{1/2}$, at which the integrated intensity of the 21.3 nm^{-1} peak is at 50% of its maximum value (a measure of when devitrification is 50% complete) vs. the heating rate for the scan. Linear regression of the three points yields an equation which can be used to estimate the critical heating rate required to shift $T_{1/2}$ above the liquidus value.

Figure 8 is the false-color pattern from Hf 105 over a broader temperature range, and at a heating rate of 1.67 °C/min. It is clear from the figure that the devitrification of Hf 105 involves more than one phase transformation, because the streaks representing the crystalline pattern are different over different temperature ranges.

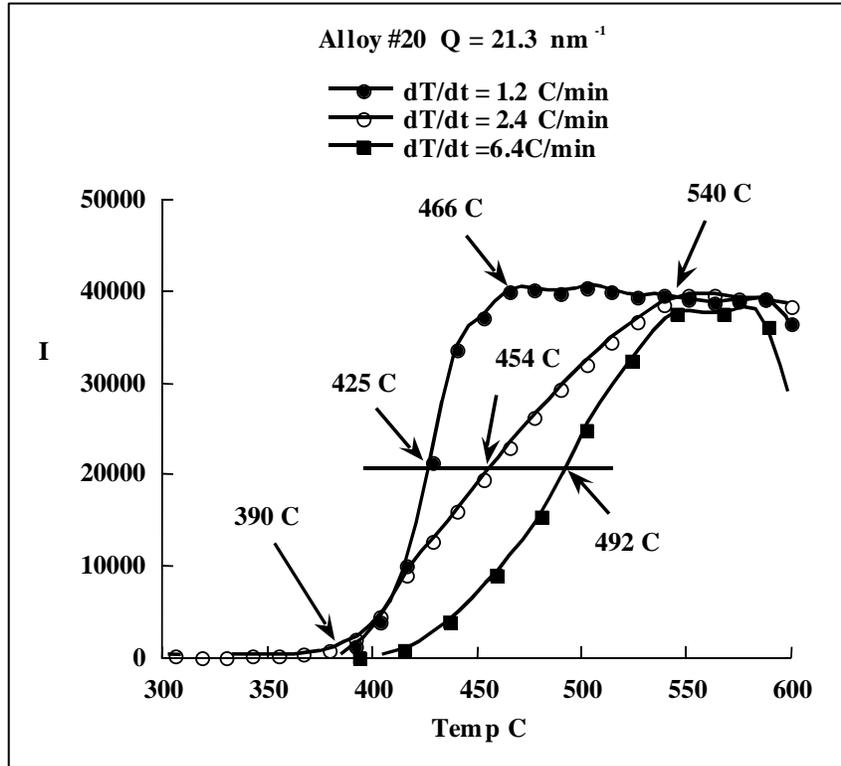


Figure 6. Plots of integrated intensity for $Q = 21.3 \text{ nm}^{-1}$ for Vitreloy 105 (also known as Alloy 20) for all heating rates. The arrows indicate the onset temperature, halfway point, and completion temperature for the transformation at each heating rate.

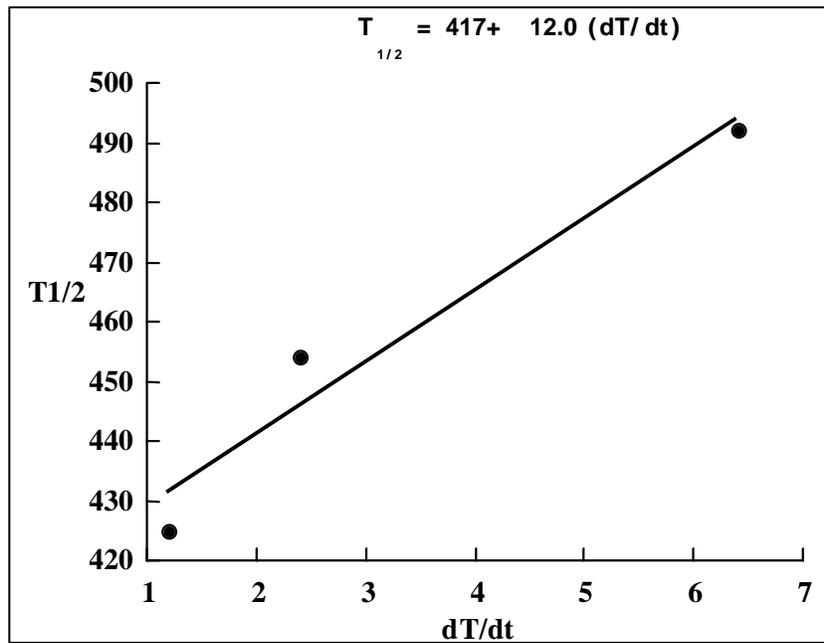


Figure 7. Plot of halfway point vs. heating rate for Vitreloy 105. The equation is from a linear regression of the three points.

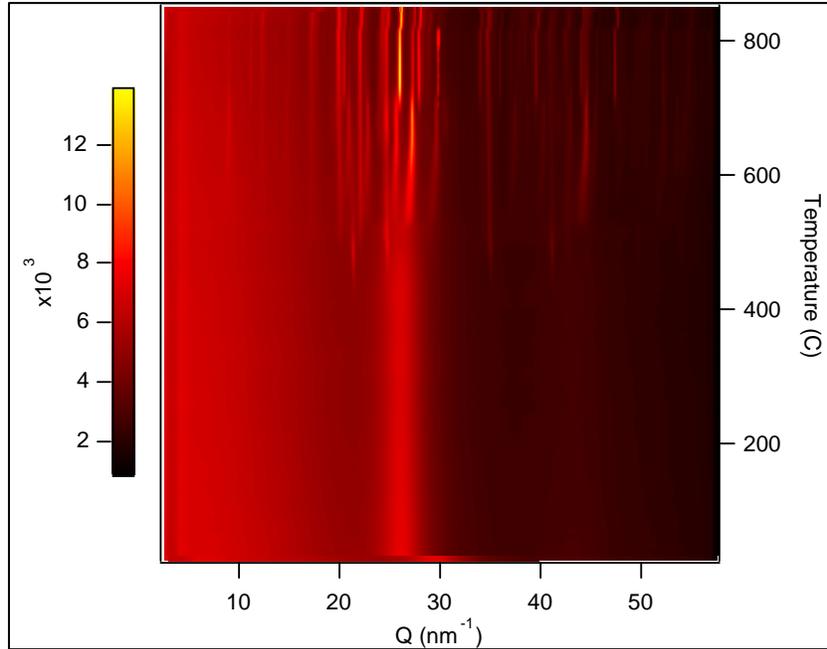


Figure 8. False-color image of XRD patterns taken from Hf 105 at 1.67 °C/min.

3.2 Critical Heating Rate

Figure 9 is a typical DTA trace used in the Kissinger-method determination of the critical heating rate. The sharp peaks correlate to exothermic events as the sample is heated. Figure 9 shows the devitrification of Hf 105 at 10 K/min. The temperature at which the peak of the first exotherm occurs is 559 °C (832 K). This is the peak used for the Kissinger analysis, i.e., the temperature at which this peak occurs is T in equations 1 and 2.

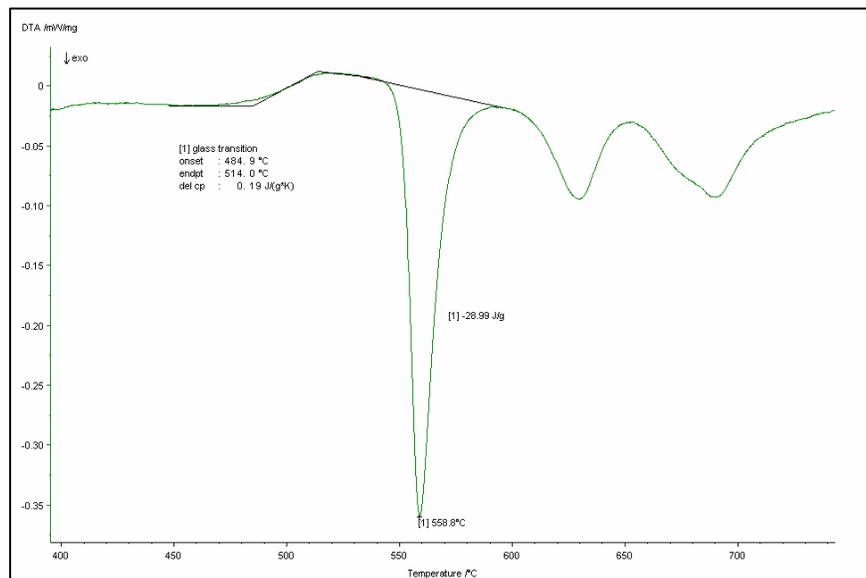


Figure 9. Typical DTA trace from $\text{Hf}_{52.5}\text{Ti}_5\text{Cu}_{17.9}\text{Ni}_{14.6}\text{Al}_{10}$.

Table 2 shows β , T , $\frac{1}{[T - T_0]}$, and $\ln(\beta/[T - T_0]^2)$ for all four tests (5, 10, 20, and 40 K/min). Using the data taken at 5 K/min, T_0 (from equations 1–3) was determined to be 515 °C (788 K). Figure 10 is the Kissinger plot. Note that the data points do not fall on a straight line. Figure 11 is a Kissinger plot that excludes the data for $\beta = 40$ K/min. This plot is linear. Using all four data points, β_{crit} was determined to be 32 K/s, or 1914 K/min. Using the three points with a linear fit, β_{crit} was 50 K/s, or 3000 K/min.

Table 2. Test results.

β (K/s)	$T - T_0$ (K)	$\frac{1}{[T - T_0]}$ (K ⁻¹)	$\ln(\beta/[T - T_0]^2)$
1/12	822	0.029455	-9.53468
1/6	832	0.022753	-9.35786
1/3	846	0.017197	-9.22466
2/3	876	0.011306	-9.37034

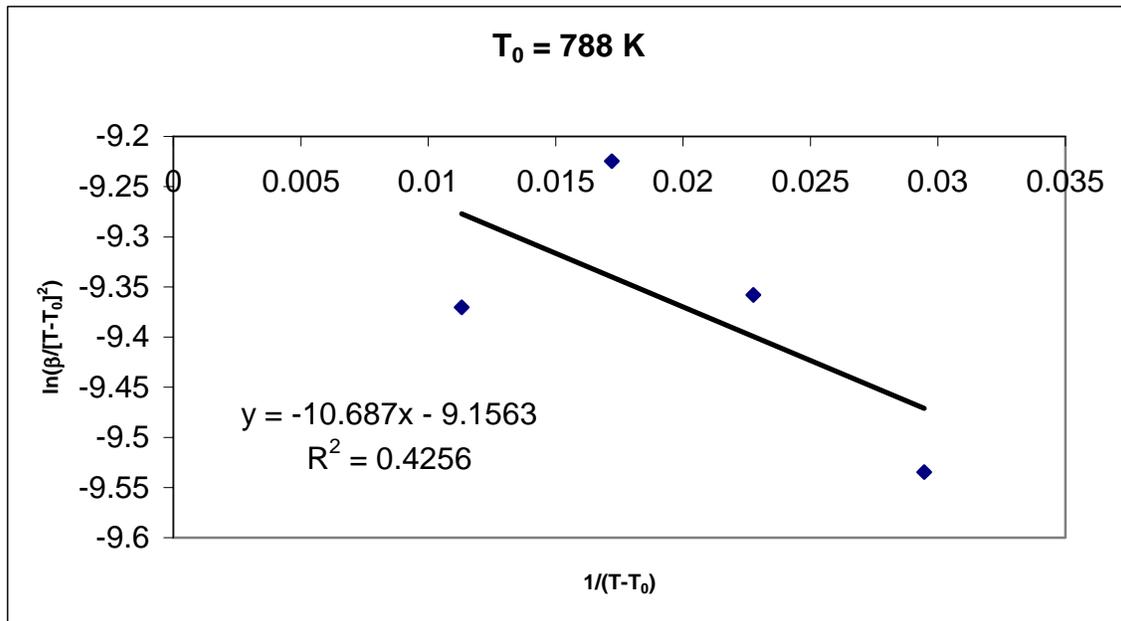


Figure 10. Kissinger plot for Hf 105 for $T_0 = 788$ K.

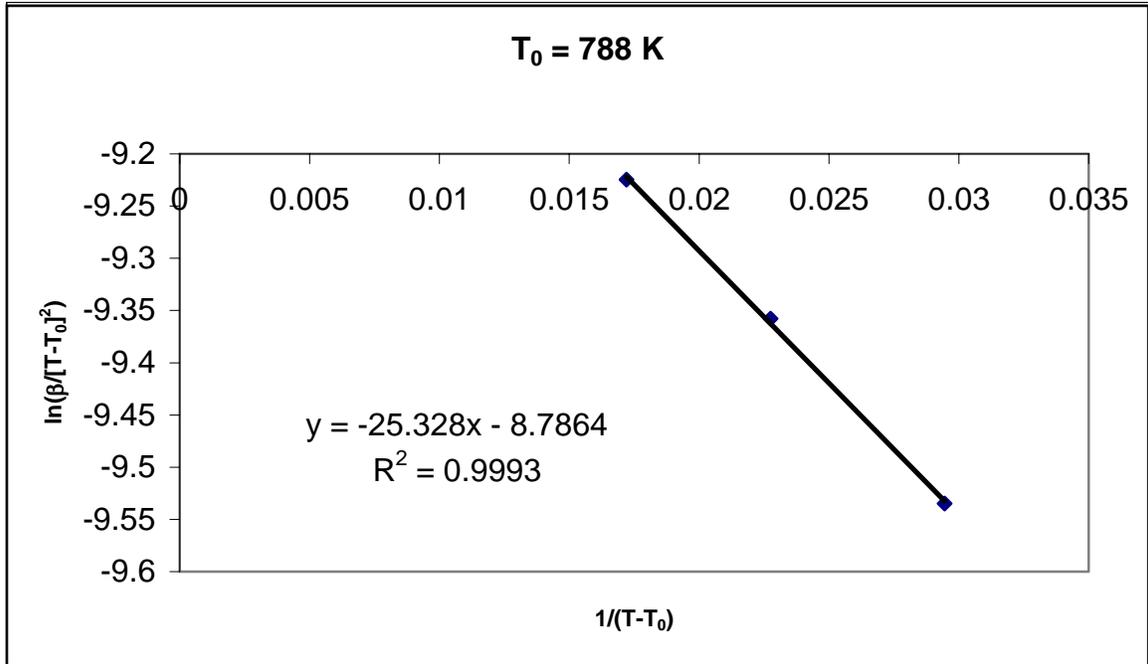


Figure 11. Kissinger plot for Hf 105, excluding the data point for $\beta = 40 \text{ K/min}$.

4. Conclusions

Heating rates of up to $12 \text{ }^\circ\text{C/min}$ are insufficient to achieve melting of typical BMGs without devitrification. All samples from which in-situ diffraction data were collected devitrified at temperatures below their liquidus. Furthermore, the results of the Kissinger-method analysis of the critical heating rate indicate that considerably higher heating rates ($32\text{--}50 \text{ K/s}$, or $1900\text{--}3000 \text{ K/min}$) are necessary to demonstrate that such melting is possible.

The implications for the ballistic behavior of tungsten-BMG composites center around the possibility that heating rates during penetration approach the critical heating rate. Given the rates involved, it seems highly unlikely that the matrix can melt without devitrification. However, it is also clear that an unequivocal experimental test of the possibility awaits further development in experimental technology.

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