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Title: The Viability of Selected Vacuum Furnace Quench Methods for Heat Treatment of Uranium

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Memorandum

NA-195 Quenching Behavior Update

Subject: The Viability of Selected Vacuum Furnace Quench Methods for Heat Treatment of Uranium

Objective:

Recent developments in high-pressure gas-quench furnaces have allowed them, in many applications, to achieve heat transfer coefficients similar to those of oil-quench furnaces. Simultaneously, high-pressure gas quench furnaces offer many attractive qualities including a smaller footprint, limited waste generation, and a more consistent quench rate across a range of temperatures. With these benefits in mind, an experimental matrix was developed to assess the feasibility of implementing a high-pressure gas quenching furnace in the Sigma facility for processing of uranium alloys. The details of this experimental matrix, as well experimental results gathered during FY21 and FY22, will be presented.

Conclusions

The results of the investigation (summarized in Figure 1) indicate that high-pressure gas quenching may not provide the necessary cooling rate for processing of uranium-based alloys such as U-6wt%Nb. In contrast, liquid-based quenching was shown to meet or exceed the required minimum 20 °C/s cooling rate between temperatures of 600 and 700 °C. Therefore, oil or water quenching remains the most effective and versatile quenching methodology for processing of uranium-based alloys.

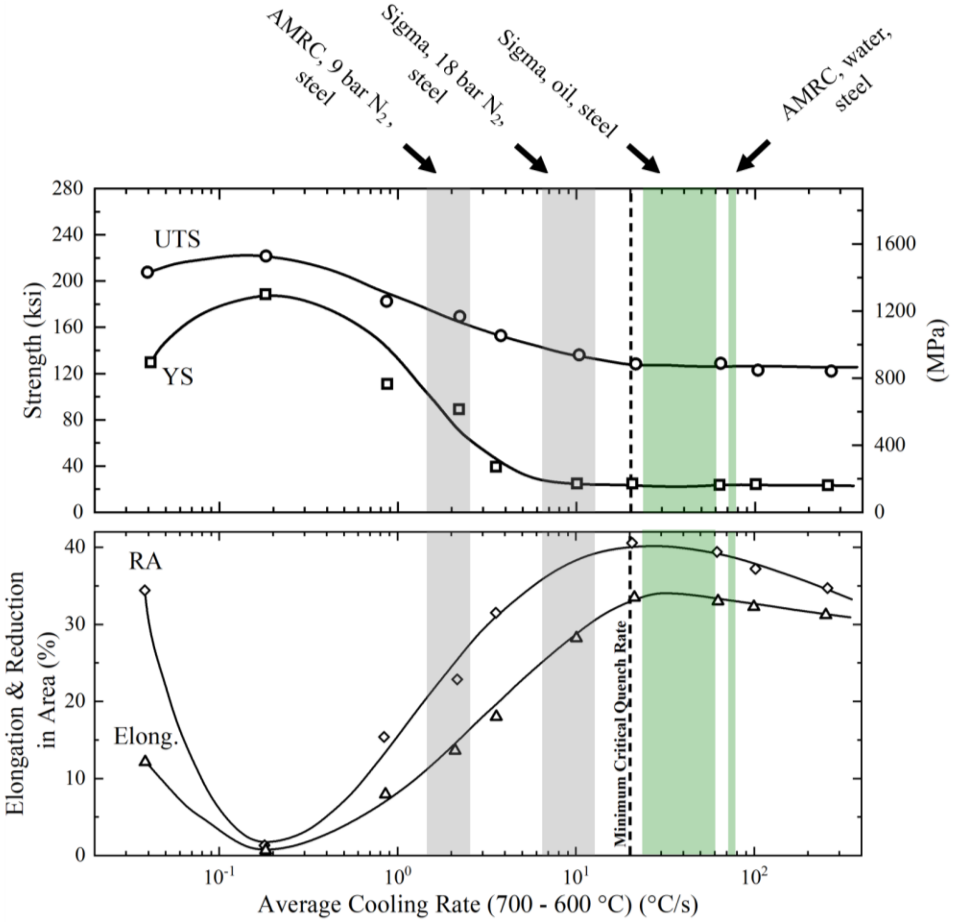


Figure 1. Cooling rates achieved using various quenchants in steels overlaid on a plot by Eckelmeyer *et al.* describing the influence of cooling rate (°C/s) on uniaxial tensile properties [1]. The critical quench rate of 20 °C/s, below which ultimate and yield strength increases with a corresponding reduction in uniform and total elongation, is indicated.

The cooling rates generated using both the gas and oil quench methodologies are summarized graphically in Figure 1, where ranges representing the mean minimum and maximum quench rate between 600 and 700 °C/s are overlaid with uniaxial tensile properties from Eckelmeyer *et al*. [1]. The minimum critical quench rate of 20 °C/s is also indicated, along with quench rate ranges corresponding to 9 bar N_2 gas quench and water quench trials completed at the Advanced Manufacturing Research Center (AMRC) at the University of Sheffield. The results of this study are summarized in document ABG3363-AMRC-19-06-20 [2]. Overall, the cooling rate ranges generated using each quenching methodology indicate that both oil and water quenching are capable of meeting minimum required quench rate for α” formation in U-6wt%Nb, while both the 18 and 9 bar N_2 gas quench furnaces produced quench rates less than this minimum. While many factors may be modified to increase the quench rate of a high-pressure gas quench furnace such as gas species, pressure, and velocity, the results of this study indicate that commercial high-pressure gas quench furnaces would struggle to provide the cooling rates necessary for processing of uranium alloys.

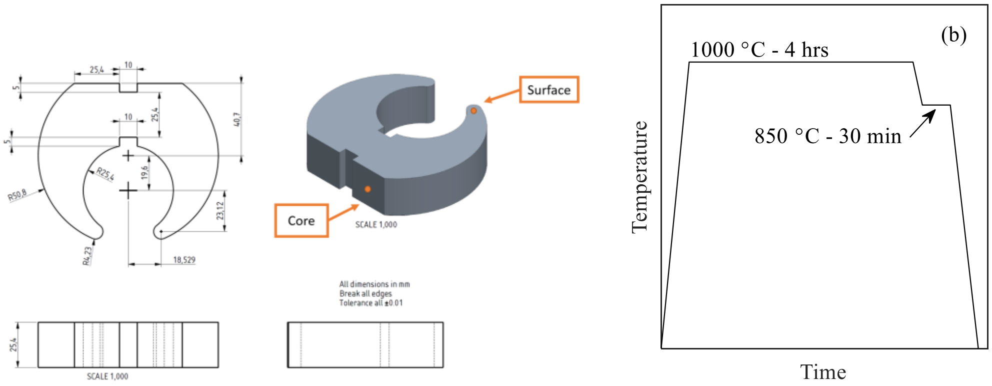
Background:

Uranium, when alloyed with elements such as niobium, titanium, and zirconium, shows a significant improvement in corrosion resistance while still maintaining a moderate level of strength and ductility. As these elements are only soluble in the high temperature γ-phase, accelerated cooling is required to keep elements in solution at low temperatures [3]. Without an accelerated cooling rate precipitation of a solute rich phase will occur, resulting in a two-phase microstructure with poor corrosion resistance and undesirable mechanical properties. The focus of this investigation is a uranium alloy with the hypoeutectic composition of U-6wt% Nb. Rapid cooling of this alloy from temperatures at or above 800 °C result in the γ-phase transforming martensitically to a supersaturated alpha phase variant, termed α”. Work by Eckelmeyer *et al.* explored the influence of quenching rate on the mechanical properties of U-6wt%Nb, with the goal of elucidating the critical quench rate to obtain a fully α'' microstructure [2]. Using various quenchants, it was concluded that for optimum mechanical properties a minimum centerline cooling rate of 20 °C/s should be achieved at temperatures between 700 and 600 °C, while for applications where only corrosion is a concern cooling rates may be as low as 2 °C/s [2].

Historically, U-6wt%Nb alloys have been processed using liquid quenchants such as oil or water, which are believed to provide the minimum centerline cooling rate of 20 °C/s between temperatures of 700 and 600 °C as the resulting microstructural and mechanical characterization is consistent with the desired α” microstructure. Recent improvements in high-pressure gas quenching furnaces may make them a viable and attractive alternative due to their smaller footprint and reduced waste generation. When comparing the two quenching media (liquid vs. gas) it is important to recognize fundamental differences in heat transfer mechanisms which result in variations in cooling rate. Due to the single-phase nature of gas quenchants cooling takes place exclusively by convective heat transfer, resulting in a single stage of cooling with across all workpiece temperatures. In contrast, utilizing a liquid quenchant results in three distinct stages of cooling, termed the vapor blanket, nucleate boiling, and convective cooling stages. These stages arise from phase changes of the liquid quenchant at elevated temperatures (boiling) and result in a variable cooling rate with workpiece temperature. For all quenching methodologies, the cooling rate is also dependent upon factors such as the specific quenching media (liquid – water, oil, polymer, gas species – nitrogen, argon, helium), quench chamber design, component size, furnace load, and load configuration.

Experimental Design & Methods:

The goal of this experiment was to determine if a high-pressure gas-quench furnace can achieve quench rates necessary for processing of alloyed uranium. As part of this experimental matrix, the quench rate of the current Wellman vacuum oil-quench furnace was characterized, as well as well as commercially available gas-quench vacuum furnace through vendor trials. A common sample geometry, shown in Figure 2(a), was utilized in all quenching trials. The selected naval c-ring geometry was manufactured in O1 tool steel; a material with similar thermal diffusivity (a measure of thermal inertia of a material) to U-6wt%Nb which could be easily provided to an outside vendor.



(a) (b)

Figure 2. (a) Naval c-ring geometry and (b) heat treatment profiles utilized in both the high-pressure gas quench furnace at a vendor facility, as well the Wellman furnace at the Sigma facility.

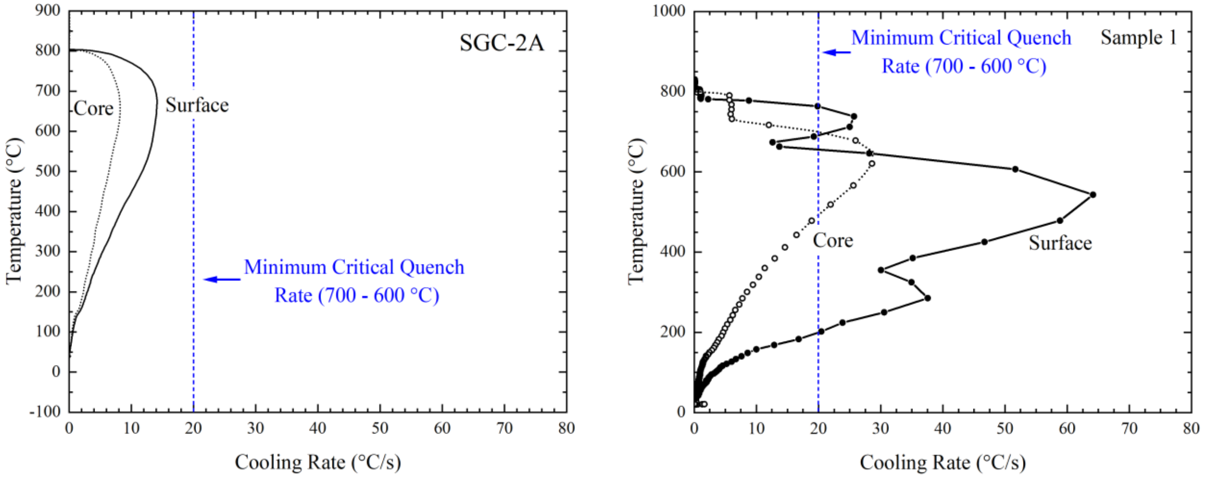
In both the oil and gas quench trials, the standard homogenization heat treatment for U-6wt%Nb, shown in Figure 2(b) was applied to three naval c-rings. High-pressure gas quenching was conducted by Seco/Vacuum in Meadville, PA in a Vector vacuum furnace type 20VP-669 as shown in Figure 3(a). Following the 30 min, 850 °C heat treatment specified for the homogenization heat treatment, quenching utilized N_2 gas at 18 bar pressure for 5 min. Thermal data from the surface and core location on each c-ring was captured using an external data logger. For oil quenching trials the Wellman dual-chamber vacuum oil quench furnace, located within the Sigma complex, was utilized where quenching occurred in a room temperature oil bath filled with Dubois Chemical AAA quench oil. Due to the unique two-zone nature of Wellman furnace a thermal barrier, specifically designed by PhoenixTM to withstand the homogenization heat treatment shown in Figure 3(b), was utilized to protect a data logger which captured thermal data from the core and surface location of each c-ring.

(a) (b)

Figure 3. (a) Vector high-pressure gas quench furnace type 20VP-669 at the Seco/Vacuum facility and (b) the Phoenix^{TM} HTS12 system assembled and loaded into the Wellman vacuum oil-quench furnace.

Experimental Results & Discussion:

Exemplar cooling curves generated from the Seco/Vacuum vendor trial and Sigma Wellman trial are shown in Figure 4(a) and Figure 3(b), respectively. The minimum critical quench rate (20 °C between temperatures of 600 and 700 °C) to ensure α” formation in U-6wt%Nb is indicated. For both quenching technologies the cooling rate curves are characteristic of the respective quench media, with the gas-quench exhibiting a single cooling rate regime while the oil-quench cooling rate exhibits three cooling rate regimes particularity at the surface thermocouple. The maximum quench rate achieved using the 18 bar N_2 quench furnaces at temperatures between temperatures of 600 and 700 °C is 14.2 °C/s at the surface and 8.2 °C/s at the core. The minimum quench rates within this temperature regime are 12.9 and 7.0 °C/s at the surface and core, respectively. The Wellman quench furnace, shown in Figure 3(b), generates greater quench rates between temperatures of 600 and 700 °C, with a maximum of 51.7 °C/s at the surface and 28.8 °C/s at the core; comparable minimums are 12.6 and 19.4 °C/s, respectively.



(a) (b)

Figure 4. Cooling rates generated using (a) the Seco/Vacuum high pressure gas-quench Vector 20VP-669 furnace (18 bar pressure N_2 gas, 5 min) and (b) the Wellman oil quench furnace with Dubois Chemical AAA quench oil.

Next Steps

The data gathered as part of this investigation will be compiled as part of a larger technical report. A continuation of this work, focused on measuring quench rates and geometric distortion of U-6wt%Nb castings during heat treatment in the Wellman oil-quench furnace, are expected to be on-going throughout FY23.

References

[1] Eckelmeyer, K. H., et al. “The Effect of Quench Rate on the Microstructure, Mechanical Properties, and Corrosion Behavior of U-6 Wt Pct Nb.” *Metallurgical Transactions A*, vol. 15, no. 7, 1984, pp. 1319–1330.

[2] “Heat Treatment Quench Trials,” Advanced Manufacturing Research Center, The University of Sheffield, ABG3363-AMRC-19-06-20, 2019.

[3] Koike , J., et al. “The Nb-U (Niobium-Uranium) System” Journal of Phase Equilibria, vol. 19, no. 3, pp. 253-260.