LA-UR-17-28727

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Intended for: Report

Issued: 2017-09-26

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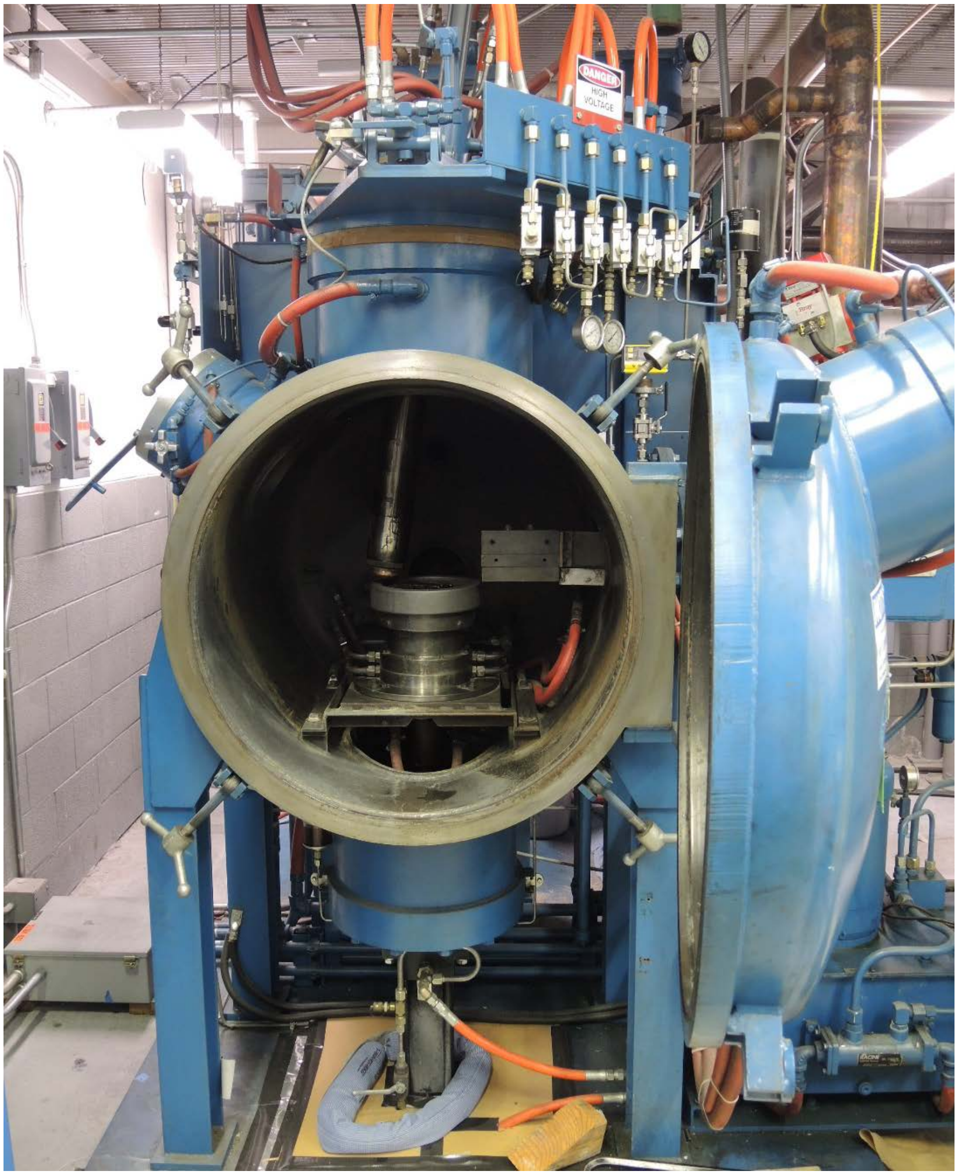

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Summary

The work described here corresponds to one piece of a larger effort to increase material usage efficiency during DU processing operations. In order to achieve this goal, multiple technologies and approaches are being tested. These technologies occupy a spectrum of technology readiness levels (TRLs). Plasma arc melting (PAM) is one of the technologies being investigated. PAM utilizes a high temperature plasma to melt materials. Depending on process conditions, there are potential opportunities for recycling and material reclamation. When last routinely operational, the LANL research PAM showed extremely promising results for recycling and reclamation of DU and DU alloys. The current TRL is lower due to machine idleness for nearly two decades, which has proved difficult to restart. This report describes the existing results, promising techniques, and the process of bringing this technology back to readiness at LANL.

1.0 PAM Use and Process Space Reduction Plan

PAM is a high temperature, dynamic melting operation with a vast process space. The current work scope attempts to reduce this process space by planning for critical variables during operation. This variable reduction is not necessarily based upon fundamental operation, but instead based upon a prioritized set of goals ordered by degree of material fully reused. These goals are: 1.) waste consolidation, 2.) recycling, and 3.) full material reclamation. To elaborate on these goals, *waste consolidation* generally means the ability to turn high surface area mixed scrap (chips, small pieces, etc.) into bulk ingots which may disposed of with an extremely high space efficiency and safer manner. *Recycling* corresponds to consolidation of scrap to a degree that it could still be reused in some operations where material quality is not of great concern. *Reclamation* would be a full recovery of either pure DU or DU-alloys to a point where they could be reused for their initial purposes.

1.1 Plasma Arc Melting Theory and Practice

The concept for plasma arc melting is relatively simple and examples of similar technologies in the form of small-scale commercial plasma cutting devices are used routinely in industrial settings. Plasma devices are most easily defined by the temperature achieved and the electrical waveform used during operation. The current project is concerned with high temperature DC plasma arcs and their interaction with the feedstock material that they are intended to melt and/or chemically modify. While extreme temperatures are the main root of a plasma system's usefulness, with a heat source that provides relatively easy access to thousands of degrees Kelvin, this thermal environment also provides a unique chemical environments that can drive reactions which are often impossible or unrealistic during other forms of processing. In the most straight forward example, these high temperatures may be used to exaggerate the differences in vapor pressure of impurity species so that only unwanted elements are removed from the workpiece being melted. Alternatively, the high temperatures and ionization of gaseous species could be used to split normally stable bonds in order to promote transient reactions at a metal surface.

A generic direct current (DC) non-transferrable arc torch, such as the one within LANL's Sigma division, is made up of a cathode, a gas injection system, and the anode. A schematic cut-away of these components is shown in Figure 1 to help put the theoretical considerations in a more practical light. This differs from the familiar non-consumable tungsten electrode arc welding instrument. In a tungsten electrode arc welder, an arc is struck between the tungsten electrode and the grounded workpiece. The arc and the electrical current flow directly between the electrode and workpiece generating the heat for melting. The plasma jet is formed independent of the workpiece and is directed toward the object being melted. A schematic and photograph are available in Figure 2 which shows the layout of moving parts in LANL's research PAM.

The gas used to generate the plasma is very important to the operation and effectiveness of melting. While the base gasses used during plasma processing are generally inert, additional gas species present within the inert atmosphere can be utilized to achieve specific arc properties [1] (e.g. temperature) which have a direct impact on both the melt pool characteristics and the reactions which may occur within the melt. The definition of inertness becomes material-dependent in this type of environment. For instance, some systems can use nitrogen as their plasma gas, but for uranium and its alloys, the stable state of uranium nitride makes this option a non-starter for recycling since the end product would be mainly uranium nitride instead of pure uranium. The ability to make chemical changes is critical for recycling applications such as the reuse of machining chips, where a wide variety of impurities may be introduced during processing. With the importance of the plasma characteristics, it is useful to record some of the theory of their formation and behavior.

Any atom or molecule may go from a stable state to an excited state. In an excited state, an atom becomes an ion. In order to reach the ion state, an atom must have an input of energy that exceeds the energy necessary to transfer at least one electron from its stable orbit to a higher energy orbit. Excitation energies are determined by the quantum states of electrons, so any collision which is lower than a minimum excitation energy simply becomes kinetic energy (i.e. increased temperature) without changing the state of the atom. A list of excitation energies for the relevant species are listed in Table 1. *Excitation* by electrical means requires an inelastic collision in which the energy is directly transferred to the energy of the atom molecule.

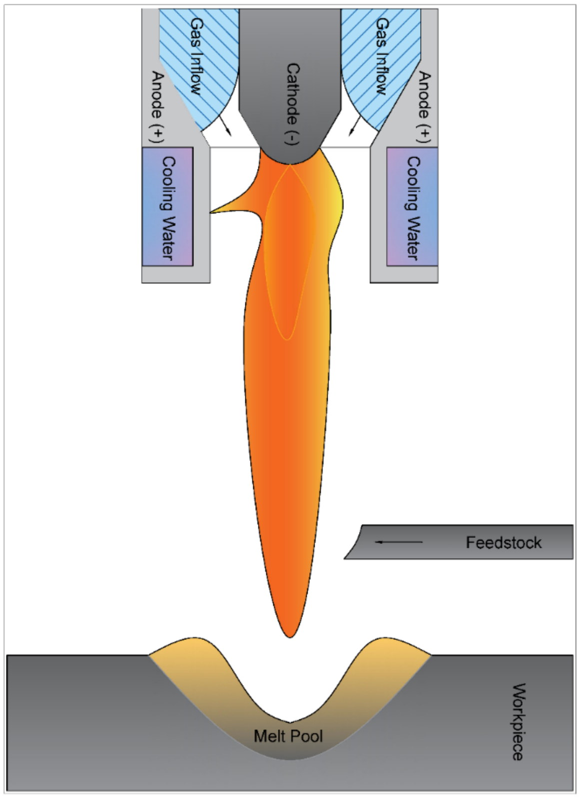

Figure 1: A schematic of a generic DC plasma arc system. Gas flows between the cathode and anode where high currents and voltages create an arc. The gas which is now a plasma is ejected from the nozzle to melt the workpiece or feedstock.

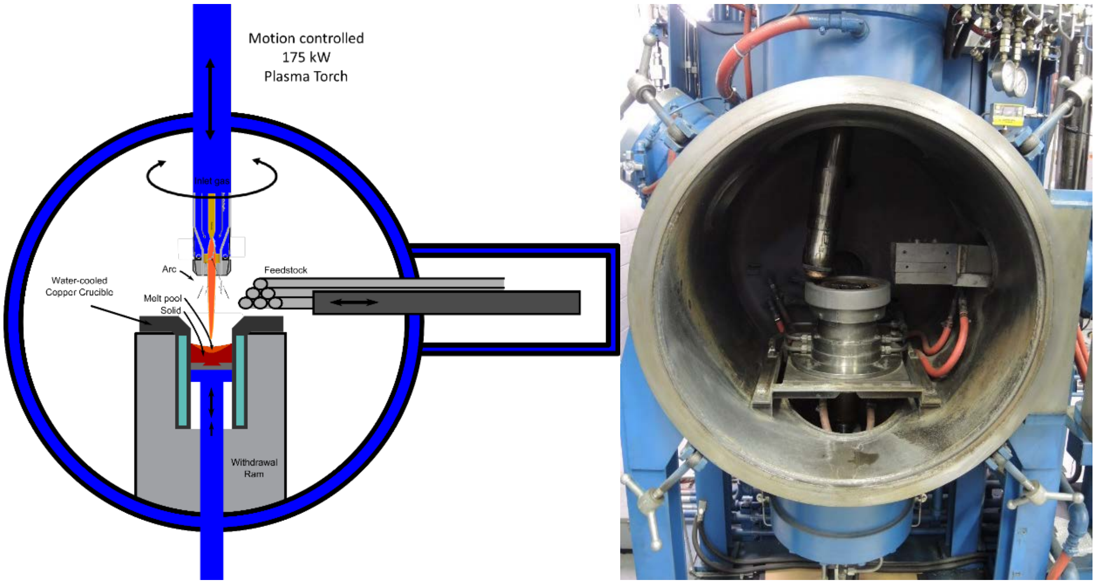

Figure 2: A schematic representation of Sigma division's research PAM compared to a photograph of the actual instrument.

Atom	Electron position	Energy (kJ/mol)
H (Hydrogen)	$2^2P_{1/2}$	983.97
He (Helium)	2^3S_1	1912.10
	2^1S_0	1997.24
O (Oxygen)	$^3P_1$	1.90
Ar (Argon)	$(^2P_{1/2})^4s$	1114.17

Table 1: Standard excitation energies for selected elements and electrons.

The excited state of an ion will always decay back to a ground state. The lifetime of the excited state is typically ~10^{-8} s, but metastable states may be attained which can prolong the lifetime of the excited state to seconds rather than nanoseconds [2]. This exact time depends upon the mean free path of the ions and the kinetic energy of the system. Alternatively, during decay, energy is released which could also excite another atom to propagate the reaction. It is these longer lived excited states (whether generated by metastable excitations, diffusion resonance transitions, or other routes to ionization) which are most useful in a PAM experiment that attempts to take full advantage of the chemical and thermodynamic space of ionized systems simply due to gas transport kinetics. For example, if the plasma generation occurs within the torch of a real system, then at a standard range of gas flow rates, it *could* take the newly created ions on the order of 1×10^{-3} s to reach the workpiece. One caveat to this is that the extreme heating of the plasma increases the plasma velocity by a factor of 10 to 100 depending on the system and parameters. However, the time to reach the work piece is still not on the order of 10^{-8} s which is a typical decay time.

Atom	Electron position	Energy (kJ/mol)
H (Hydrogen)	$^2S_{1/2}$	1138.51
He (Helium)	$^3S_1$	1910.39
	$^1S_0$	1988.54
O (Oxygen)	$^1D_2$	190.07
Ar (Argon)	$^2P_{1/2}$	1114.39

Table 2: Metastable excitation energies for selected elements and electrons.

Two of the atoms given in Table 1, H and O, exist in nature as diatomic molecules. It should be noted that the excitation energies listed there are specifically for a single atom of these elements. Excitation of molecules can also occur as well as dissociation of molecules. For instance, the molecular excitation energy of H_2 is 1487.79 kJ/mol and the dissociation energy is 1311.0 kJ/mol. This is a wonderful example because it can be seen that the dissociation energy is less than the molecular excitation energy. The reverse is true for oxygen. While the energy to achieve ionization or dissociation is the main driver for the prevalence of any of these states within a plasma, the concentrations need not be completely one-sided. In a plasma where enough energy is available to achieve any of these states for hydrogen or oxygen, the hydrogen will tend to dissociate (and perhaps ionize in the atomic form) while the oxygen will tend to ionize as a diatomic molecule. The delicate balance of these concentrations within a plasma gas can lead to an enormous array of reactions taking place even though one reaction or another may be the most energetically preferred. This concept should be kept in mind when the most relevant reactions to this project are discussed later in this report.

2.0 PAM Applicability to Material Reuse

2.1 Waste Streams

Preparing to reuse materials is the transition from a waste stream to a raw material stream. Although there are many ways of depicting waste streams, it is useful to categorize waste based upon content of organic materials, metals and oxides. Figure 3 shows a schematic representation of this categorization with examples in each of the marked regions [3]. Plasma arc melting could be used most effectively for the current range of waste issues by focusing on the metal rich areas. For machining scrap, this could fall into consolidation for less expensive disposal or a more extreme option of full reclamation. Impurity refinement feasibility depends upon the impurities involved. As discussed later in this report, it could depend upon the ratio of elemental vapor pressure more than instrumental capabilities. Casting skull is heavy in oxides and there is a possibility for oxide reduction. This isn't necessarily a recommended route, but it is technically feasible.

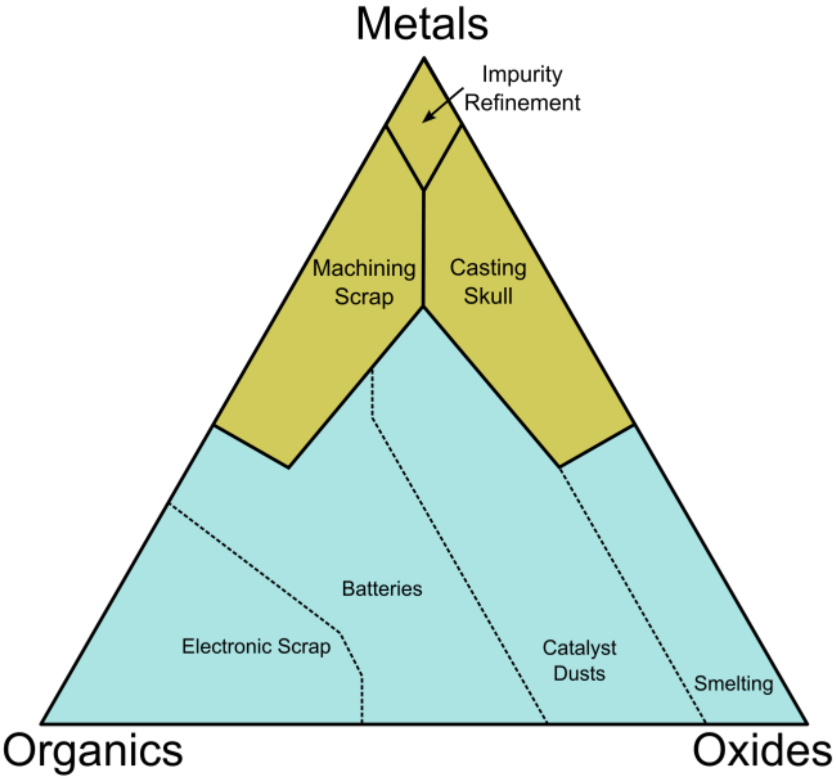

Figure 3: A schematic representation of waste classification based upon content of metals, organics, and oxides. Three categories of current interest are highlighted. Additional examples are given from other industries to provide context. Figure adapted from reference 3.

2.2 Routes to Material Modification

Material modification can take many forms. This could be a simple change in shape all the way to significant chemical changes. The extreme ends of chemical modification could be reduction of impurity level additions to purposeful alloying of metals during melting. Several of these will be discussed in this section.

Shape Consolidation

Shape and size consolidation is a critical aspect of reuse or waste disposal criteria. An example of how this is important to waste disposal is with machined metal chips. Uranium is pyrophoric, so thin metal chips or fines must be submerged in a fluid, such as mineral oil, that will prevent spontaneous combustion. However, large bulk piece of metal is not at risk for this hazard, therefore it can be safely disposed of in a much smaller volume and without additional additives. For reuse, this requires more explanation. We will start with what defines *scrap*.

Scrap materials come from processing of initial raw materials into a final part. During charge preparation, there may be machined chips generated while downsizing the starting materials. After casting, there is a mixture of oxide and metal which is termed “skull” that remains in the crucible during pouring. After casting, a part may be machined, rolled, or formed (or any combination of these). The parts of these starting shapes that do not make it to the final part are all “scrap.” Scrap can take on a broad array of sizes from very high surface area to volume chips/fines/turnings, to large hot tops. Some examples of DU-6Nb scrap are shown in Figure 4.

Figure 4: A variety of scrap materials with different shapes and sizes. Working left to right there are bundles of chips, thin rolled plate, thick picture frame, bulk hot tops.

The surface area to volume ratio is a useful value for reuse performance in casting. In general, the lower the surface to volume ratio (S/V), the higher the casting yield. We informally sampled several castings from this year and made a general comparison of yield. The results, as given in Table 3, are striking.

	Chips/Fines	Thin Plate	Thick Plate	Hot tops / Bulk
S/V	1000 to 100	100 to 10	10 to 5	<5
Yield	NA	60%-70%	70%-90%	85%-95%
Meltability	Poor	Poor	OK	Good

Table 3: An informal assessment of castings from FY17 which used charge materials with variable S/V ratios. The casting yield and meltability are noted.

Since traditional VIM casting experiences a significant decrease in melting performance with increased S/V ratios, additional routes to consolidation are required. Since there are multiple torch actions available with PAM, this difficulty can be mitigated. High S/V ratio materials can be used as raw feed stock. These can be melted directly by the plasma torch and the surface of the ingot can be periodically remelted and stirred in order to ensure that full consolidation is achieved. This is especially important with uranium alloys since the oxide is tenacious and will actually prevent melt pools from consolidating even if the underlying metal is molten.

Direct Alloying

The same features which make this technique suitable for shape modification also allow it to be used for direct alloying of materials. While other technologies could be used for large scale alloying operations, it could be reasonable to expect that some of the scrap generated during processing would require relatively minor additions of certain elements before reuse. In this case, alloying elements can be added to the feedstock in the proper ratios and the intense heat of the plasma will co-melt both the scrap and the alloying elements. The stirring action from plasma jet rotation can also provide confidence that a homogeneous product will result from processing.

Low-melting impurities

The extreme temperatures of the plasma torch allow for a chemical modification route that is largely unavailable with other technologies. In for the case of low-melting point impurities, chemical modification during normal operation is largely dependent upon the relative vapor pressure of the different atomic species (or in some cases, molecules) within the material so that some components volatilize and are removed from the system more quickly than others. Vapor pressure of different species at a single temperature may be wildly different; e.g. at 3000 K, the vapor pressure of aluminum is approximately nine orders of magnitude higher than that of tungsten. [4]

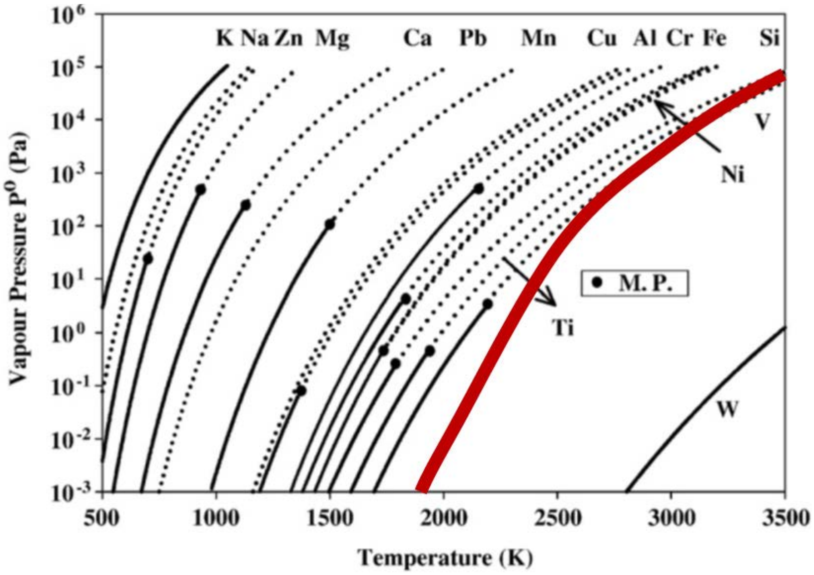

Figure 5: Relative vapor pressures of various elements with temperature. The thick red line indicates the approximate curve for uranium. This figure has been adapted from reference[4].

Temperatures inside a plasma environment can range from 5000 to 40000 K, depending upon a variety of factors. The temperature range that is actualized inside of the arc is most sensitive to the power that is applied, but the collective properties of the gas medium (e.g. thermal conductivity, ionization potential, etc.) constitute a complex but critical balance of factors. This makes atmosphere chemistry an important variable for controlling conditions during processing. The addition of non-noble gases, typically H_2, O_2, CO_2, or N_2, will lead to reactions with the specific atomic species in order to reduce oxides or to remove impurity atoms such as carbon.

Inclusion reduction

The action of the melt fluid flow dynamics coupled with predictable shape of the solid/liquid interface (which is driven by the water cooled crucible wall) provides access to another route for material improvement. Mid-sized to large impurities can become trapped against or near the mold wall. This is shown schematically in Figure 6. Pre-existing inclusions or agglomerates of particles have a different mass and move somewhat differently than the flowing liquid due to momentum transfer effects. This can result in an inclusion being driven against the mold wall where it immediately sticks within the slow moving liquid solid interface. A secondary mechanism for smaller particles is that they can become trapped between dendrites rather than pushed along toward the center of the casting.

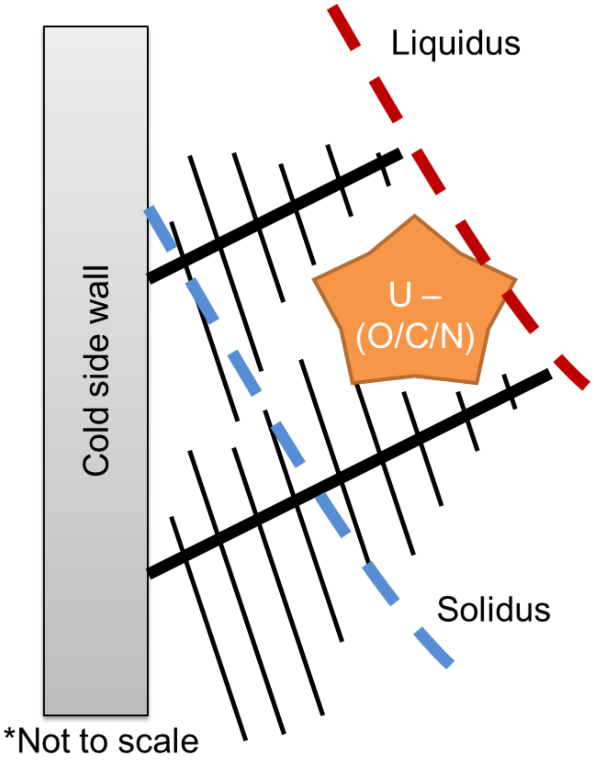

Figure 6: A schematic diagram showing inclusion entrapment between dendrites near the cold mold wall. This occurs because of local solidification dynamics as well as global fluid flow and the shape of the liquid/solid interface.

2.0 Reactions and thermodynamic stability

In a high temperature plasma environment, many chemical reactions that would otherwise be thermodynamically unlikely during other forms of processing can become possible. As was mentioned earlier in the report, while the most energetically favorable reaction will have the highest prevalence, when there is such an enormous amount of energy in the system, many types of reactions will occur. Additionally, with so many opportunities for ionization and dissociation, the number of possible reactions becomes so large that anticipating the most favorable reactions a priori becomes a difficult task at best. With that in mind, only a limited number of reactions are assessed here in order to help narrow the process space until further data can be applied for a more discriminating set of possible reactions.

Extensive thermodynamic modeling using commercial software *HSC Chemistry 7*© [5] was performed in order to determine appropriate temperature and gas ranges necessary for promotion of carbon elimination. A considerable number of these related reactions appear to become either stable or unstable at a metal surface temperature in the range of 1500-3000°C, making thermal and gas species selection (or cycling) absolutely critical for these reactions. Figure 7 shows a selection of important reactions that could be encountered and their stability with temperature.

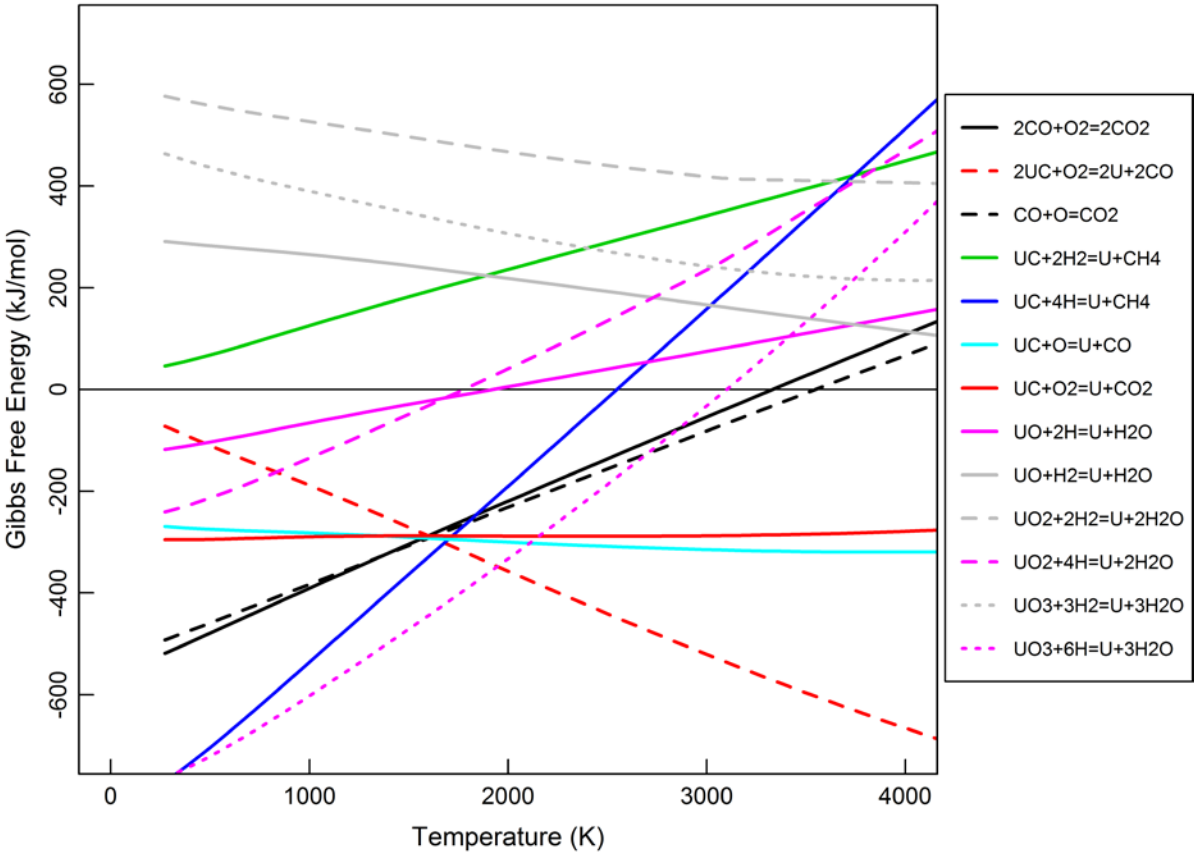

Figure 7: A selection of reactions which have been considered here in assessing the process window. Reactions are favored when the Gibbs free energy is negative.

One important consideration is the ionization and dissociation of gases within the plasma environment. Although Figure 7shows a large amount of data to easily absorb, one point to take away is that without the dissociation action in the plasma, neither the carbon removal nor the oxide reduction by hydrogen are thermodynamically possible. Using these tools, reactions which produce methane and carbon (mon/di)oxide were identified as playing a critical role (e.g. Eqs 1 and 2).

$$UC + O(g) = U + CO(g) \qquad (1)$$

$$UC + 4H(g) = U + CH_4(g) \qquad (2)$$

When the thermodynamic equations are solved for the most predominant species, it appears that even with dissociation reactions playing a role, the partial pressures that can be reasonably assumed are still insufficient to drive the reaction toward carbon removal because other reactions can dominate. That said, the concentration of species globally is difficult to ascertain and the local concentration is what matters. Any local agglomerations of oxy-carbo-nitrides for instance already locally contain the oxygen necessary to remove the local levels of carbon. As such, the problem becomes one of kinetics rather than thermodynamics.

In short, the process window for plasma arc melting appears to include space where elimination of carbon is thermodynamically preferred overall. Additionally, it has been shown that carbon removal through gas changes is possible in other alloys systems [6] as demonstrated by Figure 8. However, the competing oxidation kinetics are somewhat unknown and become critical for the final removal efficiency and total yield. This kinetic uncertainty can stretch across orders of magnitude. With uncertainty in the reaction kinetics, additional steps have been taken to provide more routes for carbon elimination during processing. Therefore, a strategy for chip pretreatment has been developed which could boost carbon elimination in the starting materials.

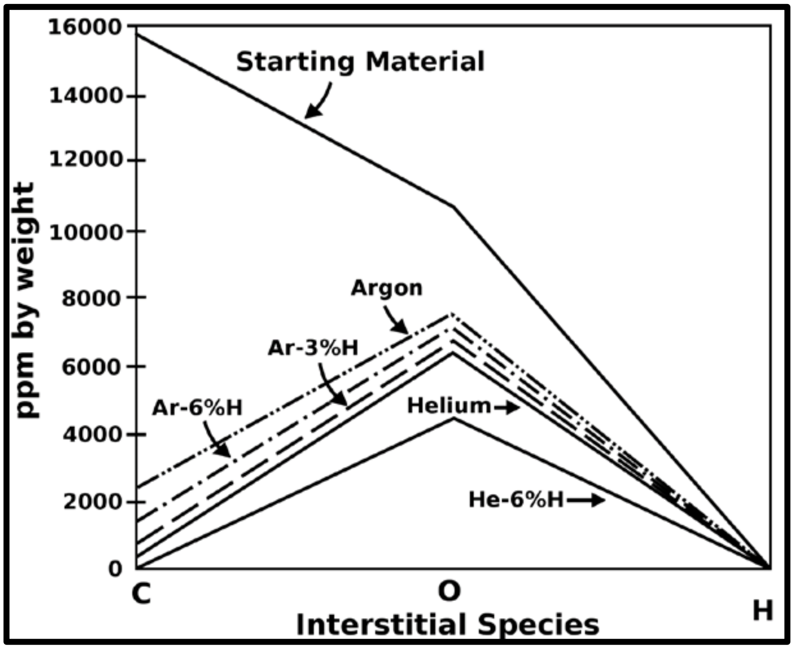

Figure 8: Carbon, oxygen and hydrogen concentration with respect to the starting concentration in Ta feedstock. Several gas mixtures are shown. Adapted from [6]

Oxidation is an easy way to remove carbon from uranium, but there is a balance to be struck since uranium also oxidizes rapidly. In order to tie oxygen to the carbide, treatment of the chips in water or steam can promote the reactions shown in Eqs 3 and 4. It is important to note that often times, a small amount of uranium nitride (UN) is present at the center of the carbide particle. Although the nitride may be important as a heterogeneous nucleation site for the carbide, the nitrogen content is generally small with respect to the carbon and the reaction product that is of interest here is the same. Therefore, the nitride will largely be ignored during discussion of these reactions.

$$UC_{(s)} + 4H_2O_{(g)} \rightarrow UO_3 \cdot H_2O_{(s)} + CH_{4(g)} + H_{2(g)} \qquad (3)$$

$$2UN_{(s)} + 8H_2O_{(g)} \rightarrow 2UO_3 \cdot H_2O_{(s)} + 2\,\mathrm{NH}_{3(\mathrm{g})} \qquad (4)$$

The reactions with the bulk uranium must also be considered (Eqs 5 and 6). The oxide formation given in eq 3 is by far the preferred reaction product. However, the kinetics of its formation appear to be inhibited with respect to the carbide reaction despite the higher driving force. One study estimated that the oxide conversion rate on the U metal was ~15 times slower than for the carbonitrides. [7]

$$U + 3H_2O_{(l)} = UO_3 + 3\mathrm{H}_{2(g)} \qquad (5)$$

$$4U + 6H_2O_{(l)} = 4UH_3 + 3O_{2(g)} \qquad (6)$$

During pretreatment, the methane and hydrogen escape from the system leaving behind hydrated UO_3 particles on the surface. It should be noted that different experiments have observed the presence of UO_3 and UO_2 depending upon specific conditions.[8]

3.) PAM Baseline Operation (DU and Alloy Charge Material)

The types of modification laid out in the previous section require baseline information concerning PAM-Metal interactions. While not all of the types of modification from above are planned for the near term, the initial experimental matrix has been created. These experiments will use bulk ingot material that is processed in a straight-forward manner with the goal of understanding chemical changes and heat transfer during melting operations. The starting material will be a cylindrical block that has been chemically sampled from the outside. The monolithic piece will serve to give fewer heat-transfer variables for analysis eventhough if it were sliced in half, chemical sampling could take place througout the bulk of the workpiece. Chemical sampling will be limited to carbon content, as this is the most important single impurity to consider for material recycling.

These workpieces will then be subjected to various processing conditions including changes in power and plasma-gas mixture. These processing variables for the experiments are given in and with the response variables that will be tracked for each condition. A schematic of the experiment and an illustration of the term “pool depth” is shown in Figure 7. The response variables from these two sets of tests will provide two broad categories of results. The first is the general heat transfer within the workpiece as given by the pool depth in combination with modeling. The second is a baseline view of chemical changes that are available within this limited process window.

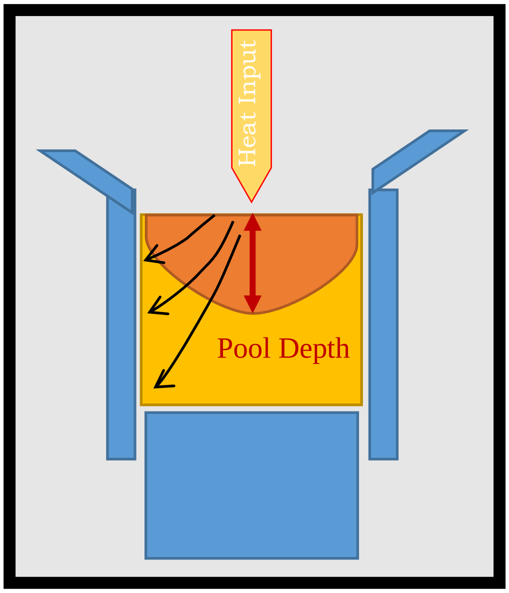

Figure 9: A schematic of the heat flow and expected pool depth shape.

Baseline Characteristics (DU)				
Power (%)	Gas (Vol.%)	Initial C (wppm)	Final C (wppm)	Pool Depth (mm)
20	Ar [100]			
40	Ar [100]			
60	Ar [100]			
80	Ar [100]			
20	Ar-H2 [98-2]			
40	Ar-H2 [98-2]			
60	Ar-H2 [98-2]			
80	Ar-H2 [98-2]			

Table 4: Unalloyed DU test matrix including conditions, process gas and responses that will be tracked.

Baseline Characteristics (DU-Nb)						
Power (%)	Gas (Vol.%)	Initial C (wppm)	Final C (wppm)	Pool Depth (mm)	Initial Nb (wt.%)	Final Nb (wt.%)
20	Ar [100]					
40	Ar [100]					
60	Ar [100]					
80	Ar [100]					
20	Ar-H_2 [98-2]					
40	Ar-H_2 [98-2]					
60	Ar-H_2 [98-2]					
80	Ar-H_2 [98-2]					

Table 5: DU-6Nb test matrix including conditions, process gas and responses that will be tracked.

DU Pucks for baseline characteristics

The DU pucks were taken from three castings. The casting IDs for these are 16C-795 and 17C-810. They were in the form of nominally 4”ODx12”L cylinders. After hot top removal, the ingot was turned down to a diameter of 3.95” to fit in the water cooled crucible of the PAM. 2” thick sections were taken (5 from 16C-795 and 5 from 17C-810). The DU in these ingots were nominally pure. The second ingot had graphite purposly put in the crucible to increase the solubalized carbon content. Images of selected test pucks and the water cooled copper crucible to be used are shown in Figure 8.

The alloy pucks were made in similar fashion. In many ways the alloy pucks are more interesting because they were made from alloy scrap material which had been rolled, cut and sheared. The nominal plate thickness was about 0.125” thick and the dimensions varied from a few centimeters across to roughly 6”x6” pieces. These high surface area pieces had been wiped down with water and acetone before processing, but were not cleaned in any other way. The first casting (17C-799) experienced an extremely low yield. The casting parameters were changed for 17C-800 to increase the yield. These were interesting because it exemplifies the problem experienced with remelting of DU-6Nb alloy. The oxide layer can be thick and tenacious. In fact, one colleague has compared its behavior to that of a water balloon. Sometimes even a pure, freshly melted surface when remelted will stretch, but not incorporate into the rest of the melt. These very thick oxide films cause a large variety of issues from low yield to large inclusions in the melt. Neither of these castings were filtered (something that LANL has done with alloy remelts for the previous 10 years).

Figure 10: The final machined pucks (foreground) from 16C-795. The PAM water cooled copper crucible can be seen in the background.

Equipment refurbishment

Although the PAM system within the Sigma Division of LANL has worked very well in the past, it did experience a long period of down time. The entire control system was refurbished and/or replaced in FY15, however, the extended down time appears to have affected systems more than was initially realized. Many of these issues could not be anticipated ahead of time.

This team has resolved each issue as it arose. However, this process of bringing the equipment back online has meant that no meaningful new data could be collected. To summarize the equipment refurbishment: This fiscal year alone, portions of the plumbing system were rebuilt, the gas injection system was modified, multiple portions of the new control system had to be adjusted, pressure interlocks were replaced, and the vacuum control system was replaced. The most damaging issue to the schedule was that the main power supply unit (175 kW, 480 V) malfunctioned. This system is old enough that the original manufacturer as well as the manufacturer which bought the first, are both defunct. After weeks of troubleshooting, the problem was resolved. However, by that time, the hydraulic system malfunctioned which controls basic movements within the furnace. Each of these issues required the system to be taken down.

The most positive part of these efforts is that the need for recycling and reuse will not diminish with time and valuable institutional equipment has been replaced part-by-part.

Communication of the Technology

The specific goals of the overarching project encompass technologies that need to be made ready for flexible and efficient manufacturing. A complex-wide effort to engage on manufacturing techniques is currently underway, so aside from the material progress of each technology, open communication with other SMEs within the NNSA complex is the most important goal of this program. Within the Sigma Foundry and Solidification Science team at LANL, the authors of this report believe that we are uniquely placed to act to progress technology, but also to fulfill the role of broad communication.

Here within LANL's Sigma Division, the activities from this fiscal year include several technologies. PAM, which this report covers directly, is a promising technology which has not been used extensively within our manufacturing network. Vacuum arc remelting is a mature technology, but still requires readiness programs. Finally, direct casting is being explored as a means of remaining flexible within a constantly changing manufacturing environment.

The Sigma division staff have played an integral role in communicating progress and pitfalls of all of these technologies. Regular conference calls give continual updates to a diverse set of SME's including staff from Y-12, LLNL and NNSA-HQ. This past February, one of the co-authors of this report organized an on-site review of technologies which included a large set of staff from across the complex as well as an independent review team. At this review, Plasma Arc Melting was introduced to many of the people there who did not realize its potential. Subsequently, Y-12 assigned specific staff members to remain up to date on these technologies.

6.0 Conclusion

The efforts from this previous year have been consolidated within this report for communication with Y-12 and other NNSA partners. A very strong suite of theoretical background and ideas for future work have been generated. While the experimental goals of this work have not been achieved due to continual "old equipment" failures, the effort has been worthwhile in the grand scheme of materials management. Any further developments in the technology will continue to be communicated to the sponsor as well as the NNSA complex at large.

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