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# Combustion Thermodynamics of Metal–Complex Oxidizer Mixtures

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**Previous chemical equilibrium calculations of metal–oxygen and metal–nitrogen reacting systems revealed that, due to the large enthalpies of vaporization–dissociation of the condensed phase formed, the flame temperature is limited to the boiling point or, more appropriately, vaporization–dissociation temperature of the products. Though it has been commonly accepted that the flame temperature of metals reacting with oxygen is indeed the vaporization–dissociation (designated the volatilization temperature) temperature of the metal oxide, the flame temperature reacting with air never achieves this volatilization temperature. Analysis of the different Ti–$N_2$ reacting systems provided the clue that, when a metal reacts with oxygen in the presence of an inert or combustion gases that do not substantially interact with the species volatilized from the condensed phase product, the enthalpy of volatilization–dissociation of the metal oxide still controls the flame temperature. Further, it has been shown that the flame temperature of a metal burning in oxygen–inert or other complex oxidizer mixtures corresponds to the volatilization temperature of the oxide evaluated at a pressure that corresponds to the partial pressure of the volatilized gases and not to the total pressure of the reacting system. This realization has important consequences in evaluating the radiative power of burning aluminum particles in solid propellants, understanding flammability limits of metal dusts in air, and applying Glassman's criterion for the vapor phase combustion of metals (Glassman, I., "Metal Combustion Processes," American Rocket Society, Preprint No. 938-59, New York, 1959), as well as in combustion synthesis.**

## Introduction

INTEREST in metal combustion had its origin in the solid propellant field when it was discovered that the addition of aluminum to composite propellants[1] increased performance. The solid oxidizer content, however, had to be reduced to maintain grain structural integrity. Further, the possibility of using liquid fuel slurries in other propulsion applications has been considered.[2] Differences between the original theoretical and experimental observations in solid propellant testing raised the question as to whether the small aluminum particles were burning efficiently; that is, did these particles burn as rapidly as liquid hydrocarbon droplets? Stimulated by the work of von Grosse and Conway,[3] who put forth the hypothesis that for metals "the combustion temperature is. . . limited to the boiling point of the oxide," Glassman,[4] realizing from droplet burning theory that the droplet approaches its own saturation temperature (boiling point), postulated that metals would burn in the vapor phase if the boiling point of the metal oxide were greater than the boiling point of the metal. If the opposite were true, then metal particles would be oxidized on their surface and not likely to be consumed efficiently. This postulate became known as Glassman's criterion for the vapor phase combustion of metals.

The essential reason for the von Grosse/Conway[3] hypothesis was that the heat of formation of the oxide at ambient conditions, which is equal to the heat release in a pure metal–oxygen system, was sufficient to raise the product oxide to its boiling point, but not to completely vaporize it. Although Glassman's[4] criterion appeared to correctly predict that Al and Mg would burn in the vapor phase and B would not, it was noted in the work of Hertzberg et al.[5] that the flame temperature of Al in air at 1 atm was not 4000 K, the oxide volatilization temperature, but 3550 K. The flame temperature results of Al and other metals reacting in air (not pure oxygen) could possibly have important consequences in the application of Glassman's criterion.[4] Although metal–oxygen–inert mixtures do not produce an adiabatic flame (combustion) temperature equivalent to the volatilization temperature of the metal oxide formed, this paper describes the fundamental reasons why the very large heat of volatility (and/or decomposition) of the metal oxide or metal nitrides still control the combustion temperature obtained. The establishment of this fact has significant importance in determining the radiative heat transfer component necessary to predict solid propellant burning rates, the lean flammability limits, and the explosivity of metal dusts and to understand some combustion synthesis processes.

## Basic Thermodynamic Considerations

As implied in the preceding section, because of the very high temperature associated with metal–oxygen reactions, it was apparent[3,4] that the oxide formed would in many instances completely or partially dissociate upon vaporization. It was known, for example, that $Al_2O_3$, the stable oxide formed in aluminum oxidation, did not exist in the vapor phase.[6] Because the thermodynamic concept of a boiling point requires that the chemical composition of the condensed phase and the vapor be the same, the question was raised[7] whether the term boiling point could be used when the condensed phase dissociated upon vaporization and, thus, whether the Gibbs phase rule did not invalidate the concept of a particular limiting temperature in metal–oxygen combustion. Detailed equilibrium calculations performed[8,9] have clearly indicated that as long as the dissociated vapor species of the oxide are in complete equilibrium the vapor species will act as a single phase.

Further, thermodynamic analyses[9] have verified that, not only do metal oxides, but also metal nitrides exhibit characteristics similar to a boiling point, and their enthalpies of vaporization–dissociation or volatilization, hitherto not thought possible to estimate, could be determined using the NASA John H. Glenn Research Center at Lewis Field CEC 80 chemical equilibrium computer program.[10] It was the insight obtained through these analyses that motivated this effort and that is the reason some review of this recent

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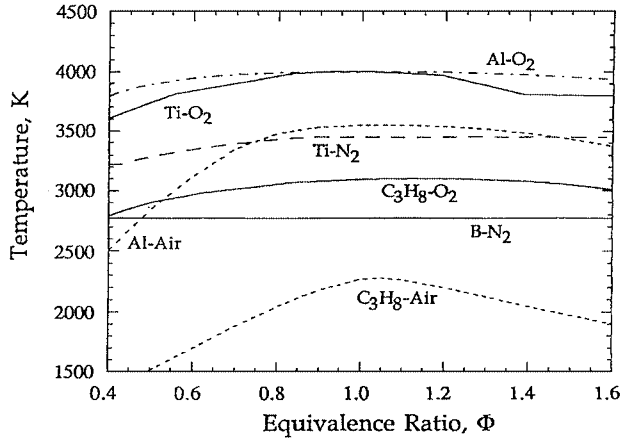

**Fig. 1 Adiabatic combustion temperature for various metal–oxidizer systems at 1-atm pressure as a function of equivalence ratio.**

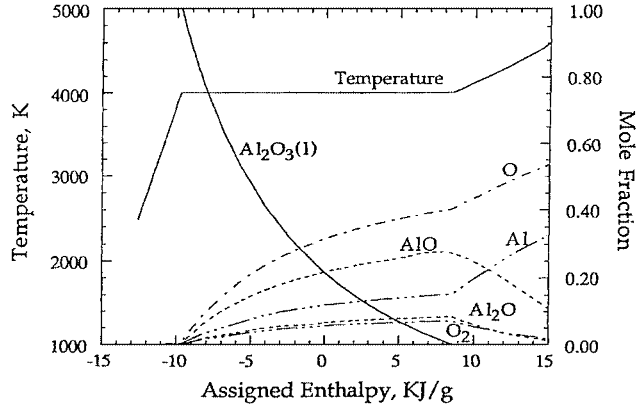

**Fig. 2 Equilibrium product composition and temperature for stoichiometric Al and $O_2$ at 298 K and 1-atm pressure as a function of the total assigned enthalpy $H_T^\circ$.**

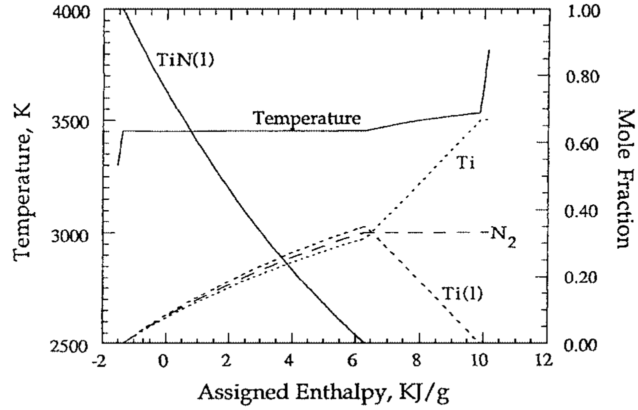

**Fig. 3 Equilibrium product composition and temperature for stoichiometric Ti and $N_2$ at 298 K and 1-atm pressure as a function of the total assigned enthalpy $H_T^\circ$.**

work is necessary to understand the physical concepts that had led to the conclusion that vaporization–dissociation characteristics of metal oxides and nitrides determine the flame temperature of many metal–oxidizer–inert systems.

Consider Fig. 1, which details the adiabatic flame temperature calculated for the oxidation and nitriding of several metals in the pure gaseous oxygen or pure nitrogen at 298 K and 1 atm as a function of equivalence ratio $\phi$. Notice that, unlike a gaseous hydrocarbon such as propane, the variation of flame temperature of many metal–oxygen or nitrogen systems with equivalence ratio is quite small. Indeed, the temperatures of the B–$N_2$ and Ti–$O_2$ systems are invariant over large regions of equivalence ratio. Of course, at equivalence ratios far from stoichiometric, the adiabatic flame temperature would drop appreciably to a value indicative of the transition temperature of other condensed phase products formed.[9] The point is that the invariance of the combustion temperatures over wide ranges of equivalence ratios substantiates the reasoning that the combustion temperature of many metal–oxidizer systems is limited due to the volatilization of the condensed phase product formed. Even with an excess of fuel or oxidizer, the energy of the reaction is always sufficient to raise the product near the volatilization temperature. This limiting temperature could be exceeded, however, if the reactants were initially at very high temperatures or large amounts of energy were added to the reacting system. Indeed, the limiting temperature also would not be reached if a sufficient amount of energy were extracted from the system. These ideas provided the method for the determination of the enthalpies of volatilization.[9]

The evaluation of enthalpies of volatilization, an important physical property of metal oxides and nitrides, was achieved by performing a series of metal combustion temperature calculations in which the assigned enthalpy $H_T^\circ$ of the reactants was varied at a given, fixed, total pressure. This procedure is analogous to varying the total enthalpy or the enthalpy of formation of the metal oxide product. The results for the stoichiometric Al–$O_2$ system are given in Fig. 2. An assigned enthalpy of zero in Fig. 2 indicates that the reactants are in their standard states at the ambient condition of 298 K and 1 atm. At this state note that the mole fraction of liquid alumina formed is 0.216. As the assigned enthalpy increases, the equilibrium temperature originally reached remains constant at 4000 K, but the amount of alumina liquid decreases. At a total assigned enthalpy of 8.56 kJ/g reactants, the condensed phase alumina is completely volatilized, and any further increase in the total assigned enthalpy will raise the temperature. Enthalpy can also be withdrawn from the system. At a total assigned enthalpy of −9.68 kJ/g, the equilibrium temperature begins to decrease, and the product composition only contains liquid alumina. These results verify that the limiting combustion temperature has the characteristics of a transition temperature or boiling point. Throughout, this temperature will be designated as a volatilization temperature. Note that for the temperature plateau in Fig. 2, the condensed phase $Al_2O_3$ mole fraction varies from 1 to 0; that is, at a mole fraction of zero, the condensed phase is completely volatilized. Thus, the extent of the overall assigned enthalpy defines the heat of volatilization, which in this case is 1860 kJ/mol. This enthalpy of volatilization (or decomposition as the case will dictate[9]) holds only for the stoichiometric condition or, to be more precise, when the ratio of metal to oxygen of the reactants equals that of the oxide in question. The value of the heat of volatilization of alumina, 1860 kJ/mol, plus the enthalpy needed to raise alumina to the volatilization temperature of 4000 K, 688 kJ/mol, is clearly greater than the overall enthalpy of reaction or the enthalpy of formation of alumina (1667 kJ/mole) at the ambient condition (1 atm, 298 K). Based on this result and others,[9] it has been demonstrated that limiting combustion temperatures are reached for many metal–oxygen/nitrogen systems.

With this background it is important to consider the same approach with respect to a Ti–$N_2$ reacting system. The results for this system are shown in Fig. 3. Compared to metals reacting with oxygen, there are particular characteristics to be noted about this system and other metal–nitrogen combustion products. First, the products are only Ti (gas and liquid) and $N_2$ as well as TiN liquid. Second, the plateau temperature rises somewhat after a given assigned enthalpy of about 6.3 kJ/g. This rise is because both Ti liquid and gas exist as products and the temperature rises rapidly when the liquid Ti is completely vaporized. For metal–nitrogen systems, although the nitride formed may disappear at a given assigned enthalpy, one of the products of the dissociated nitride may be a condensed phase. For all of the stoichiometric metal oxidation systems examined at 1 atm (Ref. 9), when the product oxide disappears, all of the products are gaseous. For the case of the B–$N_2$ system,[9] when the boron nitride completely disappears, the only products are B liquid and $N_2$ gas. Consequently, because the liquid B and $N_2$ are in their standard states at the decomposition temperature, the enthalpy of decomposition must be equal to the heat of formation of the BN, not at the ambient condition but at the adiabatic combustion or decomposition temperature. The key to understanding this point is the definition of the standard state heat of formation, which is the enthalpy of a substance in its standard state referred to its elements in their standard states at the same temperature. Because both Ti gas and liquid exist for the TiN case, the TiN products of decomposition consist of two states, and only one can be in its standard state at the decomposition temperature. Consequently, the enthalpy of decomposition will not equal the enthalpy of formation of TiN at the decomposition temperature.

When the assigned enthalpy was increased to 9.9 kJ/g reactants in Fig. 3, the decomposition products were only gases. This condition specifies a volatilization temperature of 3540 K and a partial pressure of Ti (gas) of 0.666 atm. At 0.666 atm the vaporization temperature for Ti has been found to be 3530 K. The difference between this value and the calculated vaporization temperature given by Fig. 3 is insignificant. The rise in temperature in Fig. 3 (between the assigned enthalpies of 6.3 and 9.9 kJ/g reactants) as the liquid Ti vaporizes, therefore, is due to the increase of the partial pressure of the Ti gas. Indeed, it was because this gas partial pressure could specify the combustion temperature that led to the insights of the metal–oxygen–inert results and is the purpose of this extensive introductory discourse.

## Metal–Oxygen–Inert Systems

Following the line of reasoning in the preceding paragraph, consider Fig. 4, which details the same type of stoichiometric calculation as reported for Fig. 2 (Al reacting with $O_2$) except that given amounts of inert (argon) are added to the Al–$O_2$ system. In one case, 8.46 mole of argon are added and in another 2.82 mole. Argon was chosen instead of nitrogen because gaseous nitrogen oxides would form and would slightly affect the major point to be made. The results for all conditions shown reveal a very small temperature plateau at 2327 K, the melting temperature of alumina. However, no plateau is evident during the volatilization of the alumina for the two inert addition cases. The question then arises whether the volatilization of the condensed phase product aluminum oxide controls the combustion temperature even though no plateau exists when an inert is present. Analysis of points 1–4 in Fig. 4 verifies that indeed it does. Table 1 lists the data for the explicit calculations at the four points. Points 3 and 4 are selected by considering what the gaseous partial pressures at a constant temperature (3527 K) for the two different dilution cases would be. Even though different amounts of enthalpy were assigned for these two cases, the partial pressure of the oxide dissociated gases (the total pressure minus the partial pressure of argon, $P_{\text{total}} - P_{\text{Ar}}$) are equal (0.0931 atm) in both cases. In calculating the difference between the total pressure and partial pressure of argon, only the mol fractions of gaseous species were used. The plots crossover due to the choice of units for the abscissa. The greater the amount of inert added, the less alumina is formed per gram of reacting mixture.

**Table 1 Summary data for Fig. 4**

| Point | $H_T^\circ$,[a] kJ/g | Temperature, K | $P_{\text{total}} - P_{\text{Ar}}$,[b] atm | $Al_2O_3$ (liquid), mol fraction | Ar, mol fraction |
|---|---|---|---|---|---|
| 1 | 0.0 | 4005 | 1.0000 | 0.2160 | 0.0000 |
| 2 | 0.0 | 3769 | 0.3350 | 0.1178 | 0.5868 |
| 3 | 0.0 | 3527 | 0.0931 | 0.0736 | 0.8402 |
| 4 | −3.351 | 3527 | 0.0931 | 0.2271 | 0.7009 |

[a] Total assigned enthalpy.

[b] Total assigned pressure (1 atm) minus the partial pressure of argon.

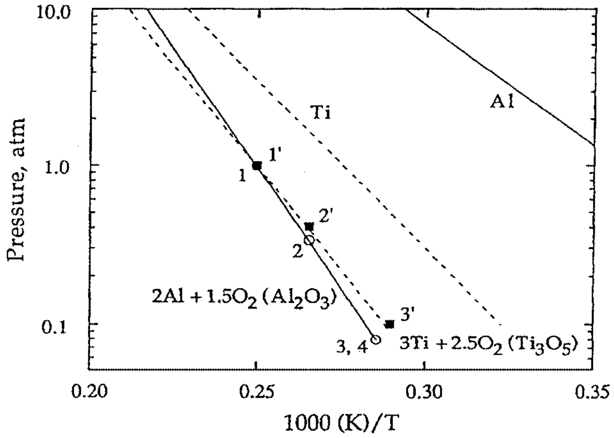

**Fig. 5 Pressure as a function of inverse vaporization temperatures of various metals and inverse equilibrium temperatures of stoichiometric metal-oxygen systems initially at 298 K.**

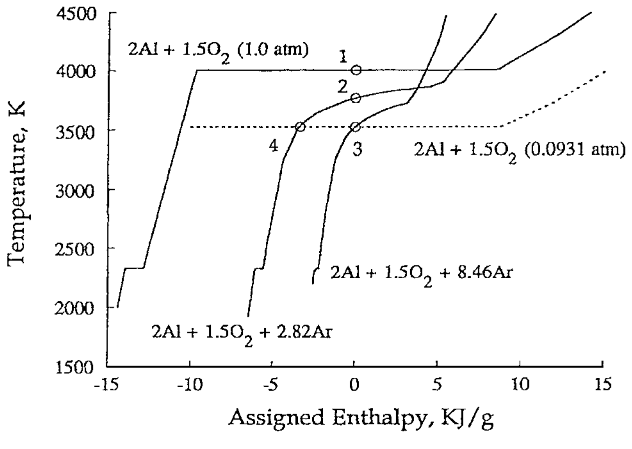

**Fig. 4 Equilibrium temperature for stoichiometric Al and $O_2$, 2Al + $1.5O_2$ + 2.82Ar, and 2Al + $1.5O_2$ + 8.46Ar, at 298 K and 1-atm pressure as a function of total assigned enthalpy $H_T^\circ$. Points 1–4 are detailed in Table 1.**

Now, consider Fig. 5, which details a plot of the calculated adiabatic flame temperature for the pure stoichiometric Al–$O_2$ system as a function of the total pressure in the form $\ln P$ vs $(1/T)$, where $T$ is the adiabatic flame temperature and $P$ the assigned system pressure. Although the lines in Fig. 5 for the oxides (metal–oxygen systems) appear straight, it can be shown over a very large pressure range that there is actually a slight curvature. This minor curvature is because the vaporization–dissociation of these oxides are not true boiling points. Whereas the lines of the elements in Fig. 5 are straight and represent the enthalpies of vaporization, those of the oxides do not.[9] For the B–$O_2$ system in which the $B_2O_3$ vaporizes congruently to its gaseous state, a plot in the form $\ln P$ vs $1/T$, where $T$ is the vaporization temperature,[9] for the B–$O_2$ system yields a straight line similar to the elements with the slope representing the $B_2O_3$ enthalpy of vaporization.

The solid line in Fig. 5 labeled 2Al + $1.5O_2$ ($Al_2O_3$) is based on calculations in which no inert had been added.[9] What is significant is that points 3 and 4 in Fig. 4 were found to have a partial pressure of 0.0931 atm for the dissociated gases: The remaining gas contributing to the total pressure of 1 atm was argon. From Fig. 5 one finds that, at a pressure of 0.0931 atm, the adiabatic flame temperature of aluminum reacting with pure oxygen is 3527 K, the same value of points 3 and 4 in Fig. 4. Similarly, the nonargon gases partial pressure for point 2 is 0.3348 atm, and the corresponding temperature is 3769 K. Point 2 ($T = 3769$ K, $p = 0.3348$ atm) also plots directly on the pure Al–$O_2$ ($Al_2O_3$) line in Fig. 5. The important significance of these correspondences is that the volatilization, or more explicitly, the enthalpy of volatilization or vaporization–dissociation determined from Fig. 2 of the condensed phase that forms, controls the flame temperature even when the reacting gaseous mixture with the metal is not pure oxygen. The Al–air temperature of Ref. 5 was recalculated to determine the vapor pressure of the nonnitrogen gaseous species. The value of the partial pressure was 0.132, which corresponds exactly to 3550 K in Fig. 5.

Figure 6 shows the data for titanium reacting with air at 1 atm, under stoichiometric conditions. The temperature–pressure variation

Table 2 Summary data for Fig. 6

| Point | $H^\circ_T$,[a] kJ/g | Temperature, K | $P_{\text{total}} - P_{N_2}$,[b] atm | $Ti_3O_5$ (liquid), mol fraction | $N_2$, mol fraction |
|---|---|---|---|---|---|
| 1 | 0.0 | 4004 | 1.0000 | 0.0923 | 0.0000 |
| 2 | 0.0 | 3768 | 0.4069 | 0.0771 | 0.5474 |
| 3 | 0.0 | 3450 | 0.1000 | 0.0661 | 0.8453 |

[a] Total assigned enthalpy.

[b] Total assigned pressure (1 atm) minus the partial pressure of argon.

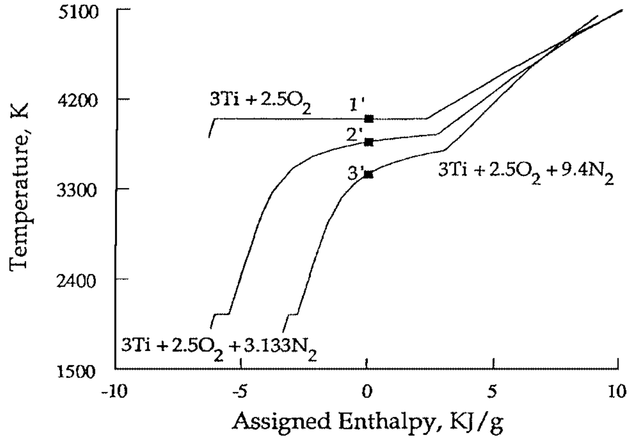

**Fig. 6 Equilibrium temperature for stoichiometric Ti and $O_2$, 3Ti + 2.5$O_2$ + 3.133$N_2$, and 3Ti + 2.5$O_2$ + 9.4$N_2$ (Ti and air) at 298 K and 1-atm pressure as a function of total assigned enthalpy $H^\circ_T$. Points 1′–3′, are detailed in Table 2.**

for the pure $O_2$–Ti system is also detailed in Fig. 5. Table 2 reports data similar to those in Table 1 for the aluminum case. Point 2′ corresponds to the adiabatic flame temperature of the $3Ti + 2.5O_2 + 3.133N_2$ system, whereas point 3′ corresponds to the case of titanium reacting with air ($3Ti + 2.5O_2 + 9.4N_2$) in stoichiometric proportions. The flame temperature of both cases are lower than that of titanium reacting stoichiometrically with pure oxygen. Point 3′ has a combustion temperature of 3450 K and a partial pressure of the nonnitrogen decomposition gases of 0.100 atm. The Ti–$O_2$ system data in Fig. 5 shows that at a pressure of 0.100 atm the adiabatic flame temperature is 3471 K, a variation of less than 1%. This difference is attributed to the nitrogen oxides formed; consequently, it is concluded that the effect of the formation of nitrogen oxides has a minimal effect on the controlling vaporization–dissociation characteristics of the condensed phase $Ti_3O_5$ that forms. When the $N_2$ for the Ti–air system was replaced with the same molar concentration of Ar, even though the specific heats of $N_2$ and Ar are greatly different, the corresponding noninert partial pressure was 0.1530 atm and the adiabatic flame temperature 3558 K. The flame temperature calculated for the pure $O_2$–Ti system at 0.153 atm is also 3558 K, and the condition falls exactly on the $P$–$T$ relationship for the Ti–$O_2$ system in Fig. 5.

These results reported for the flame temperature of aluminum and titanium burning in air have important implications. During the burning process of aluminized composite solid propellants, many aluminum particles burn in a complex oxidizer mixture. The condensed phase aluminum oxide formed in the particle flame contributes a radiative component to the propellant grain-burning rate. This radiative heat transfer must not be evaluated at the vaporization–dissociation or volatilization temperature of the oxide at the rocket chamber pressure, but, to a close approximation, at the partial pressure of the condensed oxide gaseous volatilization–decomposition components. Just as the nitrogen oxide formation had a small effect in air systems, the disturbance in the equilibrium balance of the oxide particle volatilization by the presence of other combustion gases would have a small effect on the flame temperature estimate proposed here.

Because the flame temperatures of metals in air are lower than those in oxygen and one cannot arbitrarily assume that the temperatures are equal to the volatilization temperature of the oxide at the condition pressure, the question arises as whether Glassman's[4] criterion for vapor phase combustion of metal applies when the oxidizing mixture is air. By examining the case of aluminum droplet, it appears that the criterion will most likely hold for most metals that would be used in solid propellants or whose dusts are considered explosive.[5] At 1 atm the temperature difference between Al burning in oxygen and air is 4000–3550 K (see Fig. 1) or 11%. The boiling point of pure Al at 1 atm is 2791 K. If during droplet burning it is assumed that nitrogen is the only gaseous species that reaches the droplet surface, then droplet burning theory reveals[11] that the inert concentration at a burning particle surface is given by

$$m_{i,s} = m_{i,\infty}/(1 + B)$$

where $m_{i,s}$ is the mass fraction of the inert at the surface, $m_{i,\infty}$ the mass fraction at the ambient condition, and $B$ the transfer number. The $B$ number for Al in air is about 0.77. The mass fraction of nitrogen at the surface is then 0.45. Because the atomic weight of Al is 27 and the molecular weight of nitrogen 28, then the mol fraction of Al vapor at the surface is approximately 0.55. Thus, from Fig. 5 the boiling point of Al at 0.55 atm (2665 K) is estimated to decrease by about 4.5% from its value at 1 atm (2791 K). Indeed, because the Al and $Al_2O_3$ lines in Fig. 5 diverge as one goes to lower pressure, Glassman's[4] criterion holds regardless whether the oxidizer is pure or not. Thus, under normal combustion conditions Al will burn in the vapor phase in air or pure oxygen as has been observed.

The orientation of the Ti and $Ti_3O_5$ lines shown in Fig. 5 is quite close (separated by approximately 400 K). Consequently, experimental thermal losses from the flame front would make the actual flame temperature less than the volatilization temperature of $Ti_3O_5$ to complicate matters. Indeed, combustion experiments of bulk titanium in pure oxygen[12] indicate that initially vapor phase reaction occurs next to the molten surface. As the various soluble oxides accumulate, however, the reaction zone is driven below the molten surface. Thermodynamic calculations for titanium reacting with air, as described for the Al–air system in the preceding paragraph, indicate that Ti would surface burn in air.

## Conclusions

Although the flame temperature of a metal burning in a complex oxidizer–inert mixture does not correspond to the volatilization temperature of the metal oxide at the total pressure of the particular system, the enthalpy of volatilization/decomposition of its oxide still controls the combustion temperature reached. This realization permits one to estimate an actual metal droplet flame temperature under complex combustion conditions and, thus, its correct radiative power. Over all realistic stoichiometries, the difference between the temperature corresponding to the metal oxide volatilization temperature at the system pressure and the actual metal flame temperature is of the order of 15%. Consequently, it becomes apparent that Glassman's criterion for vapor phase combustion for metals will hold for most complex oxidizing environments.

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