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Carbide Synthesis by Metal Explosions in Acetylene

SEPTEMBER 1967

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Prepared for SPACE AND MISSILE SYSTEMS ORGANIZATION
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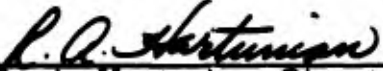
FOREWORD

This report is published by the Aerospace Corporation, El Segundo, California, under Air Force Contract No. F04695-67-C-0158.

This report, which documents research carried out from March 1967 through August 1967, was submitted on 4 October 1967 to Captain Keith S. Peyton, SMTRE, for review and approval.

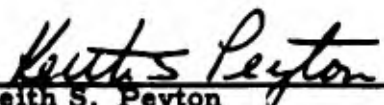
We gratefully acknowledge the assistance of Miss Cassandra Johnson, who obtained the gas chromatographic analyses.

Approved



R. A. Hartunian, Director
Aerodynamics and Propulsion
Research Laboratory

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.



Keith S. Peyton
Captain, United States Air Force
Project Officer

ABSTRACT

Thermally stable metal carbides can be synthesized readily by electrical explosion of metals into acetylene. Reasonably high degrees of purity can be obtained under appropriate reaction conditions. An important factor governing the purity of the carbide product is the necessity for dilution of the acetylene reactant with an inert gas; this minimizes the rate of self-pyrolysis of acetylene to graphite and hydrogen. It has been shown that explosive vaporization of the metal is not necessary for carbide formation since liquid droplets of the metal react readily with gaseous species derived from acetylene; thus explosive melting suffices. Using this technique, we have prepared LaC_2 , TiC , ZrC , NbC , Nb_2C , Ta_2C , MoC , Mo_2C , and W_2C from the respective metals. Of the metals studied, only cobalt failed to form a carbide. Factors involved in the synthesis of specific carbides when the metal forms more than one stable carbide are discussed.

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I. INTRODUCTION

Synthesis of inorganic compounds by explosive vaporization of metals into atmospheres of various gases is a promising technique that has not been studied very much. Such metal explosions can be created by instantaneous discharge of electrical charges stored at high potentials. Several recent studies have shown that metal nitrides,^{1, 2} XePtF_6 ,³ SF_4 ,⁴ and metal fluorides⁴ can be prepared by this technique. The SF_4 and metal fluoride syntheses were shown to be nearly quantitative if favorable reaction energetics were used. Recent experimentation in this laboratory has demonstrated, however, that the synthesis possibilities of this technique are not unlimited. Unfavorable reaction energetics and thermal stabilities can prevent the syntheses of many classes of inorganic compounds and limit the yields of successful syntheses.⁵

The present paper describes the applicability of the exploding metal method to the synthesis of thermally stable transition metal carbides. Several factors significantly influence the choice of the gaseous reactant for these syntheses. We showed previously⁴ that endothermic reactions involving metal atoms give relatively poor yields in metal explosions at imparted energy levels equal to the thermodynamic heat of reaction. This limitation applies also to exothermic processes if the reaction mechanism proceeds through a highly endothermic intermediate stage that forms a product that can be permanently stable. A relevant example of the latter phenomenon was given in Ref. 5. The extent of the pyrolysis of ethylene, initiated by metal explosions

at moderate levels of imparted electrical energies, is comparable to the extent of methane pyrolysis at similar pressures, despite the fact that ethylene, in principle, can pyrolyze exothermically to carbon and hydrogen, while the methane pyrolysis is endothermic. This can be attributed to the endothermicity of Eq. (1),



a stage that occurs in the ethylene pyrolysis to carbon and hydrogen. Due to the steep temperature drop that accompanies acetylene formation from ethylene, the resulting temperature at moderate electrical input energies is below that required for appreciable exothermic pyrolysis of acetylene to carbon and hydrogen, and the explosion product is kinetically trapped acetylene. For this reason, the synthesis of any metal carbide by the explosion of the metal in a hydrocarbon gas would be favored if the hydrocarbon were acetylene. Not only does acetylene have an extremely high endothermicity per bond, which enhances the exothermicity of carbide formation from acetylene and a metal, but acetylene cannot pyrolyze endothermically in an intermediate step to form products that are capable of permanently stable existence. This has been confirmed in our laboratory, where we have shown that with regard to carbide formation by metal explosions in hydrocarbons, acetylene is superior to olefines such as ethylene or cyclopentadiene, and the olefines are superior to saturated hydrocarbons.

The metal explosions discussed in this paper are always in acetylene, the most efficacious hydrocarbon reactant. A particular aim was to ascertain

whether relatively pure carbides could be prepared, since the self-pyrolysis of acetylene could produce a carbon contaminant. In the synthesis of Refs. 1 to 4 the gaseous reactants did not form nonvolatile contaminants.

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II. EXPERIMENTAL

The apparatus used to explode metals in acetylene was similar to that used previously for metal explosions in SF_6 ,⁴ except that a GE model 7703 ignitron switch was substituted for the mechanical Jennings switch. Furthermore, the higher energy levels obtained in the present paper require explanation. We reported previously⁴ that the discharge of three 14- μ F capacitors, connected in parallel, delivered 148 and 466 joules to the exploding metals when the capacitors had been charged to 8 and 14 KV, respectively. This represented an excessive degradation of electrical energy, since only 10.6 percent of the stored energy was delivered to the exploding metal while the remainder was dissipated in the external circuitry. The firing circuitry was subsequently redesigned to reduce these external losses. In experiments described in the present paper we were able to deliver much higher energies when three 14- μ F capacitors were charged to voltages comparable to those used previously.⁴ As previously, the delivered energies were measured calorimetrically. However, the large differences in delivered energy resulting from nominally identical electrical energy input levels demonstrates the importance of determining experimentally the actual levels of delivered energy to the exploding metals. If this is not done it is virtually impossible to compare experimental results of various investigators or to make relevant calculations of chemical energetics, since the fractions of energy degraded in the external firing circuitry can vary widely.

The metals were 20-40 mil wires. We determined the amount of wire exploded by weighing the unexploded residue, which remained attached to the

electrodes after the explosion. The extent to which the acetylene had reacted was determined by gas chromatographic analysis, on a Porapak Q column, of the volatile products of the explosion. These products were usually hydrogen and other hydrocarbons (mainly methane). From an analysis of the volatile products we could compute the fraction of acetylene converted to nonvolatile solids. These data for the titanium explosions are given in Column 5 of Table 1. From these data and the ratios of reactants we could calculate the maximum possible conversions to metal carbides, and these values are given in Column 6 of Table 1. The actual yields of carbides could be less than these maximum yields to the extent that self-pyrolysis of acetylene to carbon and hydrogen precluded the reaction of some of the Ti. The extent to which this occurred could be ascertained by elemental and x-ray diffraction analyses of nonvolatile solids scraped from the reactor wall. Carbon analyses were obtained by combustion at temperatures above 2000°C.

Column 7 of Table 1 lists the mole percents of TiC among the recovered products. These were computed from the elemental analyses. The validity of this computation was checked by the x-ray diffraction data and is discussed in the next section for individual runs. Column 4 of Table 1 tabulates the fractions of the exploding Ti that had been vaporized by the electrical input energy. These data were calculated from the quantities of exploded metal and high temperature thermochemical data in Ref. 6. Although the titanium vaporization cannot exceed 100 percent, the values cited for runs at 2188 joules are given in excess of 100 percent to indicate the actual excess energy imparted to the system in these experiments. The results are reported for the liquid and

Table 1. Product Data for Titanium Explosions

Molar Reactant Ratios ^a		Input Energy, joules	Percent Ti Vaporized by Elec. Energy	Percent Conversion of C ₂ H ₂ to Solids	Maximum Possible Yield of Carbide, percent	Mole Percent TiC in Recovered Product ^b
H ₂ /C ₂ H ₂	C ₂ H ₂ /Ti					
12.2	0.58	527	7.55	37.5	43	48
6.0	0.63	527	10.26	-	-	65
0	0.68	527	11.27	~96	100	68
12.6	0.595	2188	121.6	59	70	88
8.2	0.57	2188	109.4	65	74	94
6.0	0.60	2188	121.6	-	-	93
4.6	0.60	2188	111.6	74.5	89	84
0	0.625	2188	120.4	~96	100	65
4.7 ^c	0.64	2188	130.0	94	100	79
11.8 ^c	0.58	2188	114.1	93.5	100	93

^a0.0030 to 0.0036 mole Ti were exploded in each run, with 0.0019 to 0.0021 mole acetylene in an 800-cc volume. The Ti data reflect the amounts actually exploded rather than the weighed amounts of Ti wires.

^bCalculated from elemental analyses.

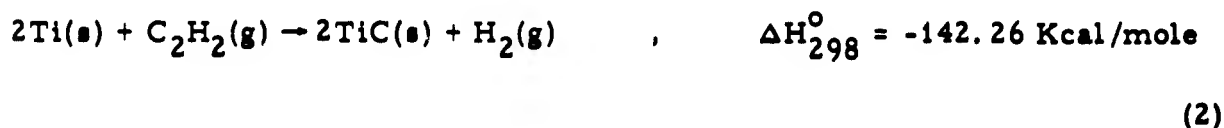
^cIn these experiments argon was substituted for hydrogen as a diluent.

vapor at 3550°K, the normal boiling point of Ti. Thus the data for the first line, for example, can be interpreted to mean that initially the exploded Ti was 7.55 percent Ti vapor and the remainder liquid Ti, at 3550°K. These results are based on the assumption that the metal wire is exploded electrically so rapidly (using an ignitron switch, probably within several microseconds) that heat exchange with the surrounding gas can be ignored initially. Clearly, these data represent only the initial states of the exploding metal. Subsequent interactions, both chemical and physical, with the surrounding gas undoubtedly lead to rapid changes in the state of the exploded material.

III. RESULTS AND DISCUSSION

A. TITANIUM CARBIDE

Attention was given first toward ascertaining whether carbides could be synthesized in high yield and good purity by the explosion technique. Analysis of existing thermochemical data indicated that the most exothermic reactions between metals and acetylene to form metal carbides and hydrogen were the reactions of the IV-A metals, titanium, zirconium, and hafnium. The stoichiometry and standard heat of reaction at 25°C for titanium carbide formation are shown in Eq. (2).⁶



Since titanium forms only one known carbide, which is extremely stable, and has a well-characterized x-ray diffraction pattern⁷ that was always observed in exploratory Ti explosions in acetylene, this system was chosen as the model system. The relevant parameter and product data are given in Table 1.

Titanium explosions in neat acetylene were very unsatisfactory. At the lower energy input level of 527 joules the acetylene reacted nearly to completion, but substantial amounts of the exploded metal did not react. Masses of unreacted metal could be clearly differentiated from the powdered carbide and graphite that lined the wall of the reactor. The x-ray diffraction pattern of the scraped product, from which the metal masses were removed,

showed the presence of large amounts of graphite and TiC and small amounts of Ti. Note that an excess of acetylene was used, above the stoichiometric amount needed for conversion of all of the exploded titanium to TiC. Reaction of 96 percent of the acetylene would thus lead to considerable carbon formation even if all of the Ti reacted. However, the carbon formation far exceeded this factor. The explosion of titanium in neat acetylene at 2188 joules, representing a 4.15-fold increase in electrical energy, gave substantially the same results, demonstrating that the poor reactions could not be attributed to a deficiency in energy. The small quantity of Ti found in the x-ray diffraction patterns of solids recovered from these runs (discrete masses of Ti had been removed mechanically) indicated that the mole fractions of TiC listed in Table 1 for these runs were probably somewhat higher than the true values.

Our explanation of the results in neat acetylene is that undiluted acetylene pyrolyzes so rapidly at these energy levels that significant amounts of graphite are formed before the Ti can react with the gaseous precursors of the graphite; such molecules could be C_2H , C_2 , or CH . Condensed droplets or particles of Ti could not react appreciably with crystalline particles of graphite, because these masses execute a rapid trajectory to the wall in the aftermath of the extremely rapid explosion. It is known that recondensation of vapor occurs very rapidly in such explosions. Note that nearly identical products were obtained in Ti explosions into neat acetylene whether the initially exploded metal was almost completely liquid or vapor.

From these data it appeared that smoother explosions leading to more complete carbide formation would occur if the acetylene were diluted with an

inert gas that could depress the rate of acetylene pyrolysis. This was proved by explosions into various mixtures of acetylene and hydrogen. At an energy input level of 527 joules, we found that the rate is depressed too much. In these runs the acetylene conversion was considerably short of completion. Titanium carbide was formed, but was contaminated with unreacted Ti and the byproduct TiH_2 , the presence of which was ascertained by analysis of the initial and final hydrogen pressures. There was no evidence for carbon formation at this energy level. Yet the extent of carbide formation exceeded significantly the fraction of Ti vaporized initially by the explosion. This demonstrates unequivocally that liquid droplets of Ti react readily in these Ti explosions and that vaporization of the metal is not really necessary. Unlike the runs in neat acetylene in which the exothermicity of acetylene pyrolysis to graphite could add energy to possibly vaporize further amounts of Ti, this was not possible in the runs now under consideration.

Better results with regard to TiC formation were obtained with acetylene/hydrogen mixtures at the higher energy level. From the data it is evident that carbides of fairly high purity can be obtained if the maximum possible yield of TiC is adjusted to be about 75 percent. At this level (and below) the contaminant is unreacted metal and possibly metal hydride. Fortunately, a considerable amount of metal plates out on the wall and cannot be recovered by scraping, further enriching the content of TiC in the recovered product. The x-ray diffraction spectra confirm that the TiC mole percents of 93 to 94, found at hydrogen-to-acetylene reactant ratios of 6.0 and 8.2 by the elemental analyses, are essentially correct within the several percent uncertainty in

the analyses and the detection limit of the x-ray diffraction method. Note that if the hydrogen dilution is lowered to the point where the maximum possible yield of carbide increases to about 90 percent, carbon formation occurs and the purity of the recovered TiC is lowered. Even with hydrogen dilution, pyrolysis of acetylene to the undesirable graphite becomes a factor if one permits the overall reaction to approach completion.

Titanium explosions were also carried out in acetylene - argon mixtures. From the data in Table 1 it is evident that argon dilutions affect the rate of acetylene pyrolysis in metal explosions to a much smaller degree than do hydrogen dilutions. In fact, we find no really detectable difference in the argon runs between extents of conversion of acetylene at Ar-to-C₂H₂ reactant mixture ratios of 4.7 and 11.8. This similarity of data appears to exceed any effect due to differences in the heat capacities of H₂ and Ar and indicates that argon is truly inert, acting merely to reduce acetylene collisions, while H₂ acts in a chemical manner; possibly this involves competing reversible reactions. We point out that one is very limited in the choice of an inert diluent for these reactions, since a number of metals react readily with N₂ in wire explosions^{1, 2} and SF₆ has been shown to react with exploding metals at energy levels considerably below those used in the present experimentation.⁴

Although argon was shown to be a milder diluent than hydrogen, it proved effective in moderating the rate of acetylene pyrolysis. There was no indication of unreacted Ti in the explosions into argon - acetylene mixtures, and a TiC sample of good purity was obtained in the run using an 11.8 ratio of reactant gases. The higher mole fraction of TiC in the latter run, as compared

to the run at a mixture ratio of 4.7, reflects only the smaller excess of C_2H_2 over the stoichiometric amount in the run producing the purer product. In these experiments, the contaminant in the recovered product was the carbon resulting from the excess C_2H_2 used as a reactant.

In summary, it has been shown that TiC can be synthesized in high yield by the exploding metal technique and that purity at least equivalent to 93-94 mole percent can be obtained. On a weight basis, which is frequently used for refractories, the purity is even higher, since 96.4 and 98.6 percent TiC by weight have been obtained when Ti and C, respectively, were the contaminants. Considerable insight was also obtained concerning processes occurring in these explosions, particularly with regard to the role of the physical state of the exploding metal and the need for a moderator of the rate of acetylene pyrolysis.

B. OTHER TRANSITION METAL CARBIDES

The next phase of this study was a survey of the applicability of the technique. This was carried out by explosion of a number of metals, at a fixed electrical energy level, into atmospheres of acetylene diluted with argon or hydrogen. Reaction parameters are given in Table 2. One observes large fluctuations in the degree of dilution of the acetylene reactant. However, this is unrelated to the efforts described in the previous section, in which specific dilution ratios were used to achieve purity of product. For the purposes of the survey of Table 2, dilution ratios fluctuated because we wanted to maintain initial gas pressures of at least 300 torr, while simultaneously varying the relative amounts of the metal and acetylene reactants. This enabled us to

Table 2. Product Data for Explosions of Various Metals in Diluted Acetylene

Metal ^a	Molar Reactant Ratio		Elemental Ratio in Recovered Product	Carbide by x-ray Diffraction
	Ar/C ₂ H ₂	C ₂ H ₂ /Metal ^b		
Lanthanum	13.5	0.94	CLa _{0.81}	LaC ₂ ^d
Lanthanum	9.4	1.17	CLa _{0.48}	LaC ₂ ^d
Zirconium	15.4 ^c	0.42	CZr _{1.75}	ZrC
Niobium	7.2	0.55	CNb _{0.69}	mainly NbC, some Nb ₂ C
Niobium	19.1	0.22	CNb _{1.66}	mainly NbC, some Nb ₂ C
Tantalum	10.1	0.95	CTa _{1.07}	Ta ₂ C
Molybdenum	5.3	1.09	CMo _{0.37}	MoC
Molybdenum	12.1	0.37	CMo _{1.04}	mainly MoC, some Mo ₂ C
Molybdenum	22.1	0.25	CMo _{1.69}	mainly MoC, some Mo ₂ C
Tungsten	21.6	0.58	CW _{3.91}	W ₂ C
Tungsten	13.6	1.01	CW _{0.93}	W ₂ C
Iron	6.0	1.05	CFe _{0.37}	probably Fe ₂₀ C ₉ or Fe ₃ C
Iron	12.6	0.28	CFe _{1.65}	probably Fe ₂₀ C ₉ or Fe ₃ C
Cobalt	9.0	0.36	CCo _{1.26}	no carbide
Cobalt	14.6	0.19	CCo _{2.84}	no carbide

^aWith the exception of the Zr explosion, which utilized an electrical energy input of 2188 joules, all other runs used 1261 joules.

^b0.0035 to 0.0045 mol of metal were exploded in these experiments.

^cH₂ was substituted for Ar in this experiment.

^dx-ray diffraction patterns obtained from Ph. D. Thesis of G. J. Palenik, Univ. South. Calif., 1960.

study the effect of reactant stoichiometry on the identity of the carbide formed by the metal explosion, since most transition metals form more than one stable carbide.

The metals selected for this survey were representative metals of Groups III-A through VI-A, as well as iron and cobalt of the Group VIII elements. Group VII-A is unrepresented only on the basis of experimental convenience, since it is difficult to obtain unalloyed wires of these metals. However, we point out that the exploding metal method need not be restricted to the use of metal wires. It is well known that metal foils or thin sheets can be electrically exploded in an appropriate apparatus.

Since the methodology of obtaining pure carbides by this technique was worked out for the model system leading to TiC formation, no attempt was made to obtain pure carbides for the metals cited in Table 2. The overall ratios of carbon to metal in the recovered products are given in Column 4 of Table 2 for the stated reactant conditions. The final column gives the identity of the carbide, as established by x-ray diffraction. Due to the variable amounts of impurities (metal or carbon), the actual carbide phase need not be directly related to the overall elemental compositions.

The most striking aspect of the data in Table 2 is the virtual independence of the identity of the carbide upon reactant stoichiometry. The carbides that form seem to be also unrelated to equilibrium energetics. This is quite different from the results of Ref. 4, in which fluorides were formed from metal explosions in SF₆. The standard enthalpies of niobium and tantalum carbide formation from the metal and acetylene are given in Table 3. From these

Table 3. Standard Enthalpies of Carbide Formation from Acetylene and V-A Metals^a

<u>Reaction</u>	<u>ΔH_{298}°</u> <u>Kcal/mole</u>
$\text{Nb(s)} + \frac{1}{2} \text{C}_2\text{H}_2(\text{g}) \longrightarrow \text{NbC(s)} + \frac{1}{2} \text{H}_2(\text{g})$	-60.6
$2\text{Nb(s)} + \frac{1}{2} \text{C}_2\text{H}_2(\text{g}) \longrightarrow \text{Nb}_2\text{C(s)} + \frac{1}{2} \text{H}_2(\text{g})$	-73.6
$\text{Ta(s)} + \frac{1}{2} \text{C}_2\text{H}_2(\text{g}) \longrightarrow \text{TaC(s)} + \frac{1}{2} \text{H}_2(\text{g})$	-61.6
$2\text{Ta(s)} + \frac{1}{2} \text{C}_2\text{H}_2(\text{g}) \longrightarrow \text{Ta}_2\text{C(s)} + \frac{1}{2} \text{H}_2(\text{g})$	-74.2

^aData calculated from thermochemical data in Ref. 6.

data one could not predict that Nb would form predominantly NbC while Ta forms Ta₂C. Similarly, the formation of W₂C, to the exclusion of WC, can not be predicted from ΔH_{298}° values of -35.4 and -19.0 Kcal/mole, per mole of metal, for WC and W₂C formation from acetylene. Clearly, we have obtained nonequilibrium behavior, in which the products formed are related directly to the temperature histories of the explosions. It is well known that transition metal carbides undergo high temperature transformations in stoichiometry. Since we have found that metal explosions in various gases frequently form kinetically trapped nonequilibrium products, one can assume that the formation of carbides other than those given in Table 2 would require a change in temperature patterns during the metal explosions, rather than changes in reactant stoichiometry. This would require either a drastic change in electrical input energy or a device for physically altering

the temperature quenching rate. Exploratory experiments in our laboratory have shown that the latter can be accomplished by attachment of the exploding metal reactor to a larger evacuated volume, separated from the reactor by a thin diaphragm. The diaphragm is ruptured by the shock wave created by the metal explosion, and one obtains temperature quenching by adiabatic expansion of the reacting species into the evacuated chamber.

Of the metals tested in this study, only cobalt failed to give an x-ray diffraction pattern of a metal carbide. This is probably attributable to the low exothermicity of its formation from Co and C_2H_2 ; ΔH_{298}° for Co_3C formation from these reactants is very much below that for the other carbides discussed above. The comparable iron carbide, Fe_3C , is somewhat more stable thermodynamically. However, while the x-ray diffraction patterns of the other carbides formed in this study were sufficiently detailed that their attribution to the respective compounds was unquestionable, the x-ray diffraction patterns of the iron explosions were not conclusive. The strongest spacings observed were at 2.08 and 2.04 Å, coinciding with the strongest spacings of the known $Fe_{20}C_9$ pattern, but many of the lesser spacings were missing. One could also observe a fragmentary pattern of Fe_3C . It is concluded that an iron carbide was formed, but we cannot definitely establish its identity.

We have thus demonstrated the general applicability of the exploding metal method to the synthesis of carbides of those transition metals that form highly stable carbides. This technique is not restricted to transition metal carbides. Although it is not included in Table 2, we have found that

Al_4C_3 is readily formed when aluminum is exploded electrically into acetylene. However, for those transition metals that form a number of carbides, the synthesis of specific carbides may require significant modifications from the simple procedure surveyed in the present study.

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13 ABSTRACT <p>✓ Thermally stable metal carbides can be synthesized readily by electrical explosion of metals into acetylene. Reasonably high degrees of purity can be obtained under appropriate reaction conditions. An important factor governing the purity of the carbide product is the necessity for dilution of the acetylene reactant with an inert gas; this minimizes the rate of self-pyrolysis of acetylene to graphite and hydrogen. It has been shown that explosive vaporization of the metal is not necessary for carbide formation since liquid droplets of the metal react readily with gaseous species derived from acetylene; thus explosive melting suffices. Using this technique, we have prepared LaC₂, TiC, ZrC, NbC, Nb₂C, Ta₂C, MoC, Mo₂C, and W₂C from the respective metals. Of the metals studied, only cobalt failed to form a carbide. Factors involved in the synthesis of specific carbides when the metal forms more than one stable carbide are discussed.</p>		

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