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# THE KINETICS OF THE THERMAL DECOMPOSITION OF MOLTEN SILVER AZIDE

by

Dr. Herbert J. Mueller

March 1967

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U. S. ARMY ENGINEER RESEARCH AND DEVELOPMENT LABORATORIES
FORT BELVOIR, VIRGINIA

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Task 1C014501A32B

March 1967

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## SUMMARY

This report covers an investigation of the thermolysis of molten silver azide ($AgN_3$), which process is considered to be fundamental for the ignition of the explosive by thermal stimulus.

The isothermal decomposition of molten silver azide was investigated in the temperature interval 309-340° C. Decomposition was found to occur at the metal-azide interface with an activation energy of $38 \pm 2.5$ kcal/mole. Initially, the decomposition proceeds at a constant rate. After a while the reaction becomes accelerated as a result of the formation of silver nuclei in the melt. The rate of reaction is then proportional to the third power of time, and an apparent activation energy of $138 \pm 5$ kcal/mole is measured. The activation energy for the growth of silver nuclei in the melt is estimated to be ca. 24 kcal/mole. The latter value can be interpreted as the activation energy for the migration of interstitial silver in the azide matrix.

## FOREWORD

Authority for the research covered by this report is contained in Task 1C014501A32B.

The study was made by Dr. H. J. Mueller, Assistant Director, Basic Research Laboratory, assisted by Messrs. R. J. Holland and C. S. Fox.

Acknowledgment is made to Dr. Z. V. Harvalik, Director, Basic Research Laboratory, for his encouragement in this endeavor.

CONTENTS


| Section | Title | Page |
|---|---|---|
| | SUMMARY | ii |
| | FOREWORD | iii |
| I | INTRODUCTION | 1 |
| II | INVESTIGATION | 1 |
| III | DISCUSSION | 7 |
| | REFERENCES | 12 |

# THE KINETICS OF THE THERMAL DECOMPOSITION OF MOLTEN SILVER AZIDE

## I. INTRODUCTION

When silver azide is brought to temperatures above 340° C, it detonates after a certain delay time. This "induction period" is a function of temperature. On heating the silver azide above 390° C, the ignition delay is very short, and the impression of a spontaneous detonation results. At lower temperatures the induction period may amount to several minutes. Microscopic observation and high-speed motion pictures show that during the induction period the azide melt bubbles vigorously, while floats of metallic silver are formed on the surface of the melt droplet. The evolution of substantial amounts of nitrogen is observed and indicates that thermolysis occurs during the initial phase of the initiation process.

The thermal decomposition of silver azide has been studied quantitatively for the solid phase only. Audubert (ref 1) calculated activation energies of 38-40 kcal/mole from nitrogen evolution measurements, as compared to 46 kcal/mole from chemiluminescence experiments. By use of the former method, Bartlett, Tompkins, and Young (ref 2) obtained 44-46 kcal/mole for the decomposition of the low-temperature form, and 31-32 kcal/mole for the high-temperature modification of silver azide. They were able to interpret the mechanism of the solid state decomposition of this salt in terms of the electron band model of the azide crystal. Silver azide, however, detonates from the melt (ref 3), and kinetic data obtained from the solid state work are consequently of limited value for an understanding of the mechanism of thermal initiation of this salt. Hence, it was the purpose of the author to study the thermolysis of molten silver azide, which process is fundamental for the ignition of the explosive by thermal stimulus.

## II. INVESTIGATION

The silver azide used in this investigation was prepared by standard techniques from highly pure silver nitrate and sodium azide and was further purified by crystallization. Colorless, prismatic crystals of silver

azide (1-5 mm) were grown from aqueous solution.[1] When these crystals were handled gently, they could be cleaved without detonating. If the crystals were allowed to age, even though they were shielded from light, they developed a faint pinkish coloration which, it is believed, was caused by the formation of silver nuclei of colloidal dispersion.[2] After the silver azide was heated, it underwent a phase transition in the solid state at 190° C.[3] The originally colorless and transparent crystals suddenly became yellow and opaque. When the crystals were recooled below the transition point, they became discolored immediately. The opaque appearance, however, remained. The opacity was probably caused by cracking of the crystals along small angle boundaries during the transition from the low- to the high-temperature modification. Molten silver azide (m.p. = 309° C)[4] is a yellow liquid of honey-like consistency, which bubbles continuously because of thermal decomposition. When the melt is brought to a temperature above 340° C, the yellow color of the droplet changes to a dark purple and detonation occurs.

Silver azide decomposes according to

$$AgN_3 \rightarrow Ag + N_2 \, . \qquad (1)$$

No secondary reactions are known, and the course of the reaction can be followed by measuring the nitrogen evolution as a function of temperature

1. The author is indebted to J. L. Taylor and R. L. Brooks, Basic Research Laboratory, for supplying silver azide crystals.

2. A similar coloration, which is well-known for aged or slightly reduced silver halides (photohalides), has been proved to have resulted from the presence of colloidal metal.

3. The low-temperature modification is orthorombic, according to Pfeiffer (ref 4). The structure of the high-temperature modification is still unknown. Preliminary x-ray measurements by J. Joebstl (private communication) indicate a rhombohedral structure.

4. Considerably lower melting points for silver azide are given occasionally in the literature. Measurements performed in the Basic Research Laboratory showed that freshly prepared pure $AgN_3$ melts at 309° C (private communication with J. Joebstl). Lower melting points are associated with impure or partially decomposed material.

and time. This method was used in the Basic Research Laboratory experiments, and the apparatus employed is shown in Fig. 1. The bottom of the reaction vessel (V) was kept at the chosen temperature by means of a thermostatically controlled metal bath (H). The nitrogen evolution was measured by a cold-cathode ionization pressure gauge (G). The output of the gauge was registered continuously by a strip chart recorder.

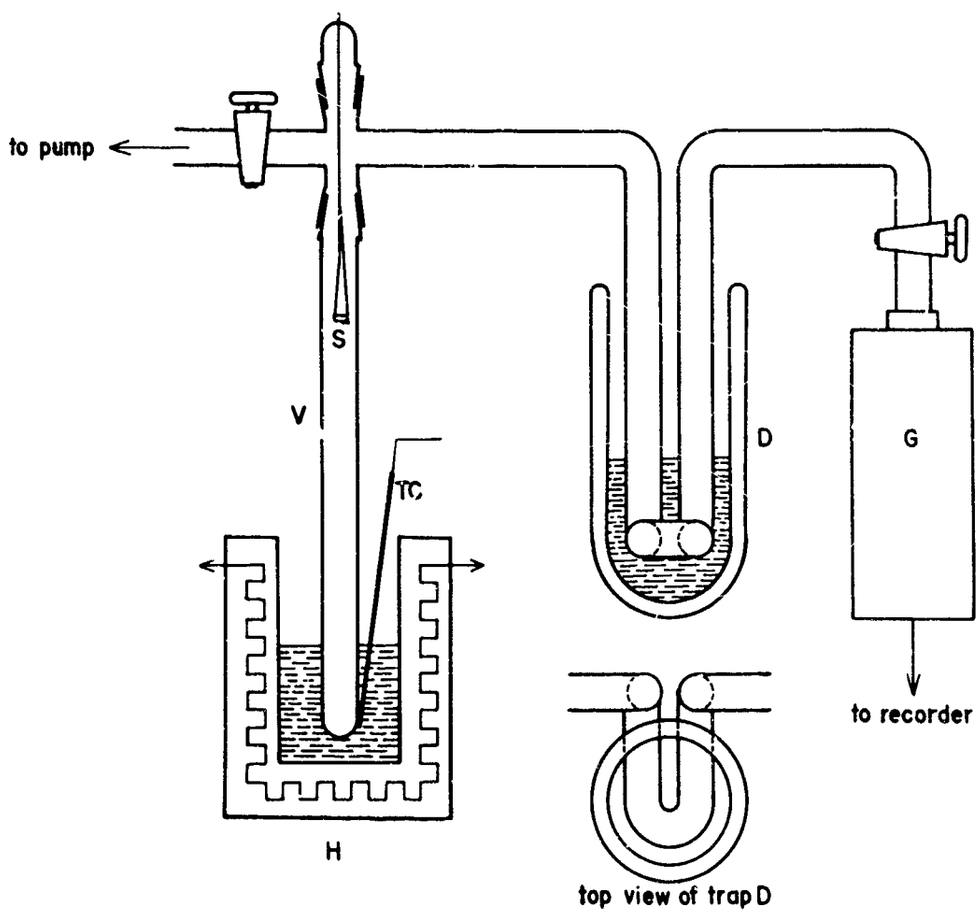


Fig. 1. Decomposition apparatus: Reaction tube (V); thermostatically controlled metal bath (H); liquid nitrogen trap (D); pressure gauge (G); magnetic sample drop device (S); and thermocouple (TC).

At the beginning of the experiment, a crystal of ca. 0.5-mg weight was placed into the sample pan of the magnetic drop device (Fig. 2). The entire system was carefully evacuated and kept at $10^{-5}$ torr for 1 hour. After this time, the reaction vessel was isolated from the diffusion pump, and the system was cryogenically pumped with liquid nitrogen for an additional 30 minutes. The measurement was started by dropping the sample crystal into the bottom of the hot reaction tube. A new pyrex tube was used for each run. The gas pressure in the system is influenced by the length of tubing that is immersed in the liquid nitrogen of the cooling trap; therefore, a special dewar (D in Fig. 1) was designed, so that the length of the cooled tubing was independent of the level of the coolant in the trap.

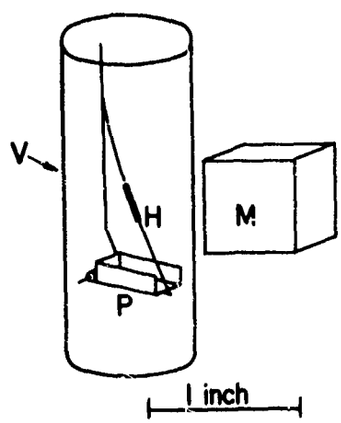


Fig. 2. Enlargement of magnetic sample drop device (S). When the steel hook (H) is released by the approaching magnet (M) to the wall of the reaction tube (V), the platinum sample pan (P) tilts downward and the specimen drops into the bottom of V.

The reproducibility of the first measurements was discouragingly poor, although utmost care was taken to use good crystals of equal weight and shape. Microscopic observation of melting crystals of silver azide indicated that the lack of reproducibility was a result of the unpredictable pattern in which the viscous, nonwetting melt spread out on a glass surface. In order to correct this situation, the bottom of the reaction tube was filled with glass beads of approximately 30-micron diameter. The spaces between the beads acted as a system of fine capillaries, into which the melt

seeped nearly instantaneously. The capillary forces caused the liquid azide to spread evenly on the glass interface. By use of this method, well-reproducible results were obtained immediately. No evidence of self-heating of the decomposing sample was found under the experimental conditions of this study.

Typical decomposition curves are shown in Fig. 3. A remarkable feature is the occurrence of two distinct patterns of reaction. When the sample is dropped, the nitrogen pressure in the system starts to build up

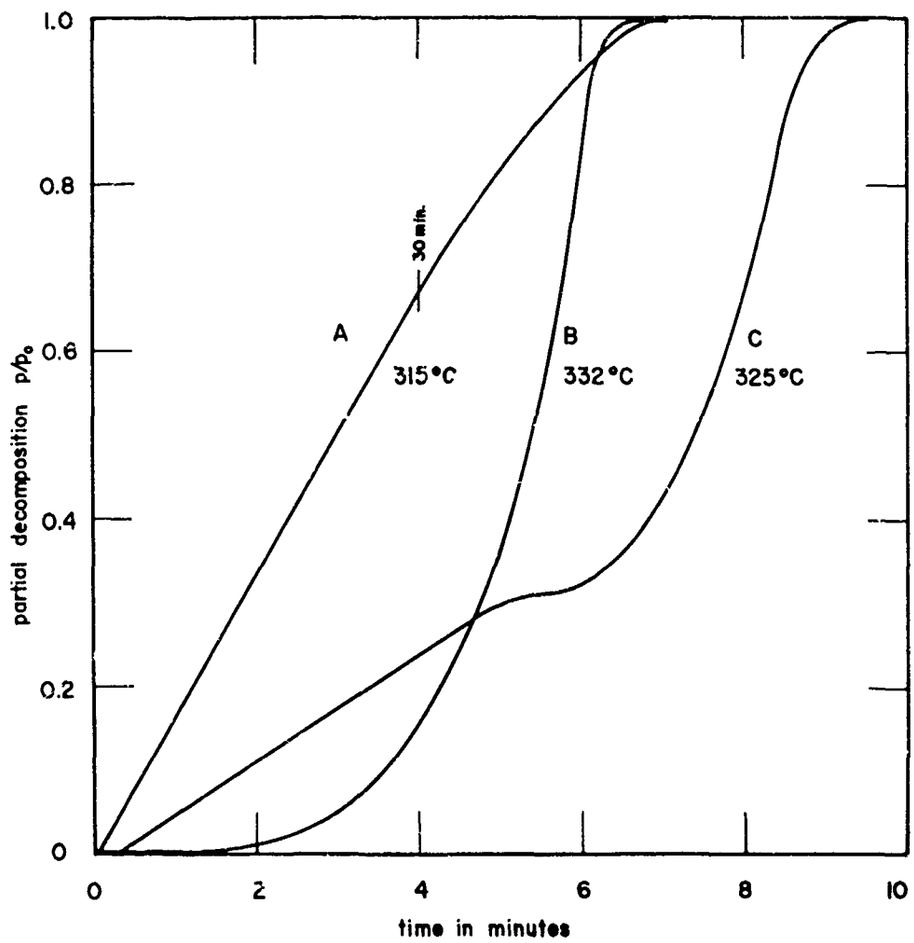


Fig. 3. Decomposition curves of silver azide. (Note the larger time scale of curve A.)

at a constant rate, and the pressure vs time diagram is a straight line. After a while, the reaction usually changes its pace and becomes accelerated. The onset of the acceleration occurs earlier at higher temperatures. The linear branch may shrink to a minor irregularity of the decomposition curve at the beginning of the experiment. On the other hand, the linear reaction persists without any acceleration when the sample temperature is not too far above the melting point.

The linear branch of the decomposition is typical for a first-order reaction, which takes place at an interface of constant area. From the Arrhenius plot, an activation energy of $\varepsilon_I = 38 \pm 2.5$ kcal/mole was obtained for this phase of the reaction (Fig. 4). Microscopic observation of the decomposing sample showed that immediately after melting of the specimen, the glass-azide interface is covered with thin silver flakes, and floats of metallic silver appear on the free surface of the melt. Gas

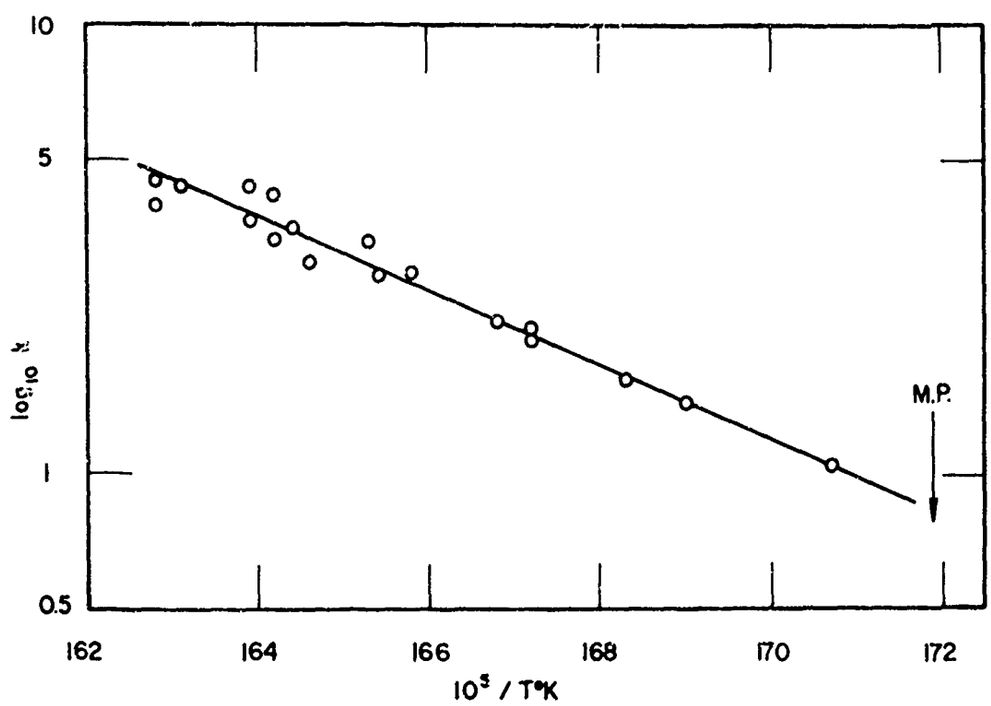


Fig. 4. Arrhenius plot for the thermolysis of silver azide (time-linear reaction, k in arbitrary units).

bubbles were observed to rise from silver particles only; thus, it must be concluded that decomposition occurs predominantly at the silver-silver azide interface.

## III. DISCUSSION

If the onset of the accelerated reaction is taken as zero time, and if the total pressure background produced by the linear reaction is properly subtracted, the pressure buildup (P) during the accelerated phase of the reaction can be plotted as a power of time, with an exponent of four

$$P_{accel.} \sim t^4 \tag{2}$$

The exponents in individual runs have been found to vary between 4.2 and 3.7; the lower values are predominantly observed at higher temperatures. It was impossible to calculate the activation energy for the accelerated reaction with some accuracy from "mixed" curves (C in Fig. 3) because of the difficulty of determining exactly the new zero point of the time axis. Experience showed, however, that freshly prepared crystals occasionally gave decomposition curves of type B also at relatively low temperatures. Hence, a number of crystals of equal weight from one particular batch were selected and were decomposed either at 326°, 329°, or 332° C. Only those curves which showed the $t^4$-law from the beginning were considered for evaluation. The specific rates obtained from these runs were averaged and used for an Arrhenius plot from which an apparent activation energy of $\epsilon_{II} = 138 \pm 5$ kcal/mole was finally computed.

Power laws (e.g. as represented in equation (2)), with exponents between 2 and 8 frequently have been found to govern the thermolysis of metal azides (ref 5). These laws have been interpreted in terms of formation and growth of metal nuclei and resultant acceleration of the thermolytical reaction. The occurrence of a $t^4$-law, in particular, is indicative of a first-order reaction which takes place on the surface of three-dimensional, steadily growing metal nuclei, while new nuclei are being formed constantly. According to the theory, the apparent activation energy $\epsilon_{II}$ then consists of three times the activation energy for one-dimensional nucleus growth $\epsilon_G$, plus the activation energy for the formation of new nuclei $\epsilon_F$

$$\epsilon_{II} = 3\,\epsilon_G + \epsilon_F\,. \tag{3}$$

Generally, it is complicated to analyze $\epsilon_{II}$ numerically, because both $\epsilon_G$ and $\epsilon_F$ are usually unknown. For molten silver azide, however, a reasonable value for $\epsilon_G$ is at one's disposal. Obviously, the activation energy $\epsilon_I$ for decomposing the azide ion at a compact metal surface--a process which results in the formation of metallic silver--must be identical with $\epsilon_G$, the activation energy for the one-dimensional growth of a compact silver nucleus. Substitute $\epsilon_I$ for $\epsilon_G$ in equation (3). Thus, one arrives at approximately 24 kcal for $\epsilon_F$, the activation energy for the formation of new metal nuclei.

The physical significance of $\epsilon_F$ is as follows: Because ionic transport phenomena in heteropolar silver salts usually involve the transport of interstitial silver ions only, the basic process of nuclei formation in silver azide can be assumed to be the migration of excess silver ions to potential sites of nucleation, such as vacancies or impurities. Thus, the probability of forming new metal nuclei will be determined essentially by the number of potential sites, the number of excess (interstitial) silver ions, and the mobility of these ions in the azide matrix. The migration of interstitial ions in a lattice is governed by an activation energy of

$$\Phi = \frac{W_1}{2} + W_2 \tag{4}$$

where $W_1$ denotes the energy for promoting an ion from its regular site to an interstitial position, and $W_2$ is the activation energy for cations to move from one interstitial position to another. Assume that the energy required for the silver ions to associate with the potential sites of nucleation is negligible. Then, the temperature dependence of the nucleation process will be given by $C \cdot \exp[\Phi]$, and $\Phi$ should be identical with $\epsilon_F = 24$ kcal/mole in equation (3). If the argument is valid, one can expect that other transport phenomena in silver azide which depend on interstitial silver ions should have the same temperature dependence as the formation of metal nuclei. In fact, Young (ref 6) measured an activation energy of 1.08 eV, or approximately 24.8 kcal/mole, for the temperature dependence of the ionic conductivity of silver azide. This value is in good agreement with the corresponding value of $\epsilon_F = 24$ kcal/mole from the decomposition experiments made in this study.

The physical meaning of an activation energy of 38 kcal/mole for the metal-azide interface reaction in the molten phase is not clear as yet. Bartlett, Tompkins, and Young related the activation energy for the solid

state decomposition to the energy necessary for thermal formation and subsequent dissociation of excitons $N_3^*$. In the absence of optical data on molten silver azide, such as the absorption spectrum and the ratio of the static to the high-frequency dielectric constant, and because of the lack of structural information on the melt, it is hardly possible to assess the validity for the molten silver azide of a similar exciton concept at the present time. On the other hand, it is interesting to note that the activation energies for the thermal dissociation of strictly covalent azides are very close to those measured for molten inorganic azides, such as $AgN_3$ and $KN_3$ (as shown in the table). The thermal decomposition of covalent azides is known to follow the pattern

$$RN_3 \rightarrow RN + N_2 \qquad (5)$$

$RN_3$ is considered to resonate equally among the structures $R\text{-}\ddot{N}\text{=}\overset{+}{N}\text{=}\underset{\cdot\cdot}{N}:^{-}$ and $R\text{-}\overset{-}{\underset{\cdot\cdot}{N}}\text{-}\overset{+}{N}\text{≡}N:$ (ref 7), and the activation energy for thermolysis is essentially the dissociation energy of the bond between the first and second nitrogen of the azido group.

Activation Energies for Thermal Decomposition of Azides

| Compound | ε (kcal/mole) | Reference |
|---|---|---|
| methyl azide | 40.8 | * |
| | 43.15 | 8 |
| ethyl azide | 40.1 | 9 |
| | 39.7 | 8 |
| n-propyl azide | 39.4 | 9 |
| iso-propyl azide | 38.5 | 9 |
| silver azide (molten) | 38 | this paper |
| potassium azide (molten) | 39 | ** |

* M. S. O'Dell, private communication.
** H. J. Mueller, unpublished work.

The occurrence of an activation energy of 38-39 kcal/mole for molten azides raises the question to what extent a mode of decomposition given by equation (5) is possible in inorganic azide melts. Even though silver azide shows some covalency in its bond character, it has a prevalently ionic structure, and thus, a massive volume reaction, according to equation (5), cannot be expected. Experimental evidence, however, reveals that this reaction or one closely related is possible for inorganic azides at a metal interface. Clusius and Schumacher (ref 10) studied the electrolytical decomposition of an aqueous solution of isotopically center-labelled potassium azide, $K^{14}N^{15}N^{14}N$, as well as the thermolysis of this salt, and found that the species $^{15}N_2$ was practically absent in the decomposition products. They concluded that dissociation of the azide occurred by bond fracture $^{14}N - {}^{15}N^{14}N$. This, however, is essentially the decomposition pattern of organic azides, and an activation energy of the same magnitude can be expected for both organic and inorganic azides which decompose in the liquid phase at a metal surface. The experiments reported herein show that this is actually true.

Little can be said as to the intermediate steps of the melt decomposition. The presence of a metal interface in the reaction is essential for discharging the azide ions. Subsequent unimolecular decomposition of the azide radicals is unlikely because the easiest permitted reaction requires 62.3 kcal/mole (ref 11) as compared to the experimentally observed 38 kcal/mole. A ground state dissociation with only 7.5 kcal/mole is spin-forbidden, but could be of importance for decomposition in pairs via an $N_6$-intermediate. There is no evidence for the existence of such a species, however. The results obtained by Clusius and Schumacher, as well as the fact that the activation energies for organic and molten inorganic azides are practically identical, suggest that in both types of azides the decomposition must occur under similar configurational and energetical conditions, that is, the azido group must be attached to some sort of a radical by a covalent bond. With a metal interface, such a covalent bond could be established by chemisorption, and the following reaction steps at a metal speck $[Ag_x]$ are feasible:

$$[Ag_x] + N_3^- \rightarrow [Ag_xN - N_2]^-. \qquad (6)$$

This first step amounts to the polarization and chemisorption of the azido group at the metal surface, while an essentially covalent surface bond is formed. When the necessary activation energy is available, bond fission occurs and gaseous nitrogen evolves:

$$[Ag_xN - N_2]^- + 38\ \text{kcal/mole} \rightarrow [Ag_xN]^- + N_2. \qquad (7)$$

The remaining nitrogen radical may migrate on the surface and combine with another radical, followed by desorption of $N_2$. The excess charge of the metal speck becomes finally compensated by a silver ion:

$$[Ag_x]^- + Ag^+ \rightarrow [Ag_{x+1}]. \qquad (8)$$

This reaction scheme would explain well the experimentally observed values of the activation energies, the $N$-$N_2$ bond fracture being the rate-determining step for the thermolysis of both organic and molten inorganic azides. Further investigations, however, will have to prove the foregoing hypothesis.

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13 ABSTRACT

This report covers an investigation of the thermolysis of molten silver azide ($AgN_3$), which process is considered to be fundamental for the ignition of the explosive by thermal stimulus.

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