DEVELOPMENT OF PEX EXPLOSIVES
SOLID-LIQUID PHASE DIAGRAMS OF ENERGETIC MIXES

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DEVELOPMENT DIVISION

JULY - SEPTEMBER 1971
SANL 900-009

For
Lawrence Livermore Laboratory
Livermore, California

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DEVELOPMENT OF PEX EXPLOSIVES:

SOLID-LIQUID PHASE DIAGRAMS OF ENERGETIC MIXES

ABSTRACT

The solid-liquid phase diagrams of various binary mixtures of FEFO, BDNPA, EDNP and BDNPF are being studied. Temperatures at which a 0.025-inch probe penetrated the samples were measured.

It appears that the samples had not crystallized but solidified into a glass.

INTRODUCTION

A study of the solid-liquid phase diagrams of various binary and ternary energetic mixtures is being carried out. The compounds of interest in this study are bis(2,2-dinitro-2-fluoroethyl)formal [FEFO], ethyl 4,4-dinitropentanoate [EDNP], bis(2,2-dinitropropyl)formal [BDNPF], and bis(2,2-dinitropropyl)acetal [BDNPA]. Thermal analysis is being used to determine the solid and liquid phases present at each composition and temperature from -54 to 74 C.

Previous attempts at determining the phase diagrams of these materials using DTA were unsatisfactory. Two other thermal methods have been tried thus far in the present study. Solid-liquid phase diagrams of many systems can be determined by measuring the changes in slope of plots of temperature versus time(1).

A slower rate of cooling is obtained while a solid phase is separating out because the heat evolved by solidification partly offsets the heat lost by radiation and conduction to the surroundings. Detection of the transition from solid to liquid was also attempted by Thermal Mechanical Analysis (TMA). In this method the temperature at which a 0.025-inch probe penetrated the sample (~ 0.060 inch thick) was measured. Neither of these experiments has given the desired results.

PROCEDURE

A simple apparatus was used for obtaining the cooling curves of various mixtures of FEFO, EDNP, BDNPF, and BDNPA. Approximately 10 ml of each mixture was contained in a jacketed tube and immersed in a dewar of liquid nitrogen. The out-

(1) F. Daniels, et al. Experimental Physical Chemistry, McGraw-Hill Book Company New York 1970.

put of a Chromel-Alumel Thermocouple inserted in the sample was plotted as a function of time. The sample was stirred with a wire.

The penetration measurements were made on the Du Pont DTA (Model 900) using the thermal mechanical analysis attachment. The instrument was calibrated using compounds of known melting points. A sharp break was observed for these materials at the melting temperature. The sample thickness (~ 0.060 inch) and sensitivity was chosen to give a full scale deflection for complete penetration of the sample. The samples were melted in aluminum DSC sample cups. They were then solidified by quenching on dry ice. The temperature was raised from -120 C to $+40$ C at 10 C/min. The total time that any sample was maintained at a low temperature was less than 10 minutes.

RESULTS AND CONCLUSIONS

No freezing points were obtained from the cooling curves. The temperatures from the penetration measurements are given in Tables I through III for binary mixtures of BDNPA-BDNPF, BDNPA-FEFO, and BDNPF-FEFO. These temperatures are unrealistically low since the temperatures for pure BDNPA, BDNPF, and FEFO are not in agreement with the reported melting points(2). It appears that the samples have not crystallized, but solidified as a glass. The temperatures measured would then be the glass transition temperatures and not the melting point of the materials.

The melting points of pure BDNPF and BDNPA have been measured using the hot stage microscope and they are in agreement with the values reported in reference(2). Crystallization of these samples was induced by seeding the supercooled melt. It is probable that the mixtures used in the other experiments can be induced to crystallize if they are given adequate time.

FUTURE WORK

Additional experiments are to be conducted in an attempt to determine the solid-liquid phase diagrams of energetic mixes of BDNPA, BDNPF, FEFO, and EDNP. Thermomicroscopy will be used to determine the essential characteristics of the temperature-composition diagrams. In order to use this method the low temperature range of the Leitz hot-stage is being extended to -68 C. Additional penetration measurements and DTA experiments are to be made on samples which have been stored at -54 C long enough for crystallization to occur.

(2) G. Repp & R. Squire, LLL Report UCRL - 7969, October 7, 1964.

Table I. Penetration Temperatures for Mixtures of BDNPF in BDNPA

<u>% BDNPA</u>	<u>% BDNPF</u>	<u>T C</u>
100		-45
90	10	-45
80	20	-46
70	30	-46
60	40	-45
50	50	-46
40	60	-46
30	20	-45
20	80	-46
10	90	-45
0	100	-47

Table II. Penetration Temperatures for Mixtures of BDNPA in FEFO

<u>% FEFO</u>	<u>% BDNPA</u>	<u>T C</u>
100	0	-70
90	10	-65
80	20	-58
70	30	-61
60	40	-57
50	50	-54
40	60	-54
30	70	-50
20	80	-48
10	90	-46
0	100	-45

Table III. Penetration Temperatures of Mixtures of BDNPF in FEFO

<u>% FEFO</u>	<u>% BDNPF</u>	<u>T C</u>
100	0	-70
90	10	-65
80	20	-63
70	30	-59
60	40	-58
50	50	-55
40	60	-54
30	70	-51
20	80	-50
10	90	-48
0	100	-47