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DEPARTMENT OF DEFENCE
DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION
MATERIALS RESEARCH LABORATORIES
MELBOURNE, VICTORIA

REPORT

MRL-R-916

**THE EFFECTS OF IMPURITIES AND ADDITIVES ON THE
GROWTH OF POLYCRYSTALLINE TNT**

William S. Wilson

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ABSTRACT

Growth of polycrystalline TNT on thermal cycling depends upon the presence of impurities in the TNT. Growth increases with the concentrations of 2,4-dinitrotoluene and 2,3,4-trinitrotoluene individually (these materials are major impurities in commercial TNT), and these materials have a synergistic effect on growth when they are present together. On the other hand paraffin wax and beeswax, commonly added as desensitisers to TNT-based compositions, have no effect on the growth process. Finally the presence of anthracene and 1-nitronaphthalene, additives recommended to reduce exudation and/or cracking in commercial TNT charges, increases growth in polycrystalline TNT. These results are fully in accord with the mechanism proposed previously to account for the growth process.

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The effects of impurities and additives on the growth of polycrystalline TNT

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ABSTRACT

Growth of polycrystalline TNT on thermal cycling depends upon the presence of impurities in the TNT. Growth increases with the concentrations of 2,4-dinitrotoluene and 2,3,4-trinitrotoluene individually (these materials are major impurities in commercial TNT), and these materials have a synergistic effect on growth when they are present together. On the other hand paraffin wax and beeswax, commonly added as desensitisers to TNT-based compositions, have no effect on the growth process. Finally the presence of anthracene and 1-nitronaphthalene, additives recommended to reduce exudation and/or cracking in commercial TNT charges, increases growth in polycrystalline TNT. These results are fully in accord with the mechanism proposed previously to account for the growth process.

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THE EFFECTS OF IMPURITIES AND ADDITIVES
ON THE GROWTH OF POLYCRYSTALLINE TNT

1. INTRODUCTION

Many items of Australian ordnance are filled with TNT-based explosives such as Composition B (RDX/TNT/Beeswax 55/45/1), taking advantage of the low melting point of TNT (80.8°C) to allow the fillings to be melt-cast using steam-heated equipment. However, certain properties of TNT also place restrictions on the use of these compositions. Isomeric dinitrotoluene and trinitrotoluene impurities form low-melting eutectics (m.p. as low as about 37°C) with the major constituent 2,4,6-trinitrotoluene; these eutectics may migrate to free surfaces of TNT-based charges as an undesirable liquid exudate [1]. Similarly the weak, brittle nature of TNT and its tendency to solidify from the melt as large, elongated crystals make such compositions prone to cracking, which has been implicated in premature detonation on gun firing of projectiles filled with TNT-based compositions [2]. The strength of TNT can be increased and its susceptibility to cracking can be alleviated by causing the TNT to solidify in an array of small randomly oriented crystals. Such an array is produced in creamed TNT, prepared by casting a slurry of molten and solid TNT poured at the melting point of the explosive, or when a small quantity of hexanitrostilbene (HNS) is incorporated in the TNT in a special casting procedure [3].

Polycrystalline charges of TNT are also dimensionally unstable, being subject to irreversible expansion on thermal cycling below the melting point of TNT. This irreversible expansion occurs in all directions and results in an increase in volume of the charge. It is commonly termed growth. Growth of TNT-based compositions can clearly have undesirable results, particularly in thinly cased devices in which the explosive contributes to structural integrity.

It has been shown elsewhere that growth of RDX/TNT compositions depends on both the number of thermal cycles to which the charge is subjected and the maximum temperature experienced, and that growth is linked with the anisotropic thermal expansion of TNT [4,5]. Investigations at MRL have confirmed that growth of creamed TNT increases with the number of cycles, and

increases linearly with the maximum temperature of the cycle, provided it exceeds a critical temperature, ca 37°C. It was shown that growth also depends on the presence of the isomeric dinitrotoluene and trinitrotoluene impurities. The extent of growth correlates with the quantity of these impurities; creamed purified TNT without the impurities experiences no growth when cycled up to 60°C. Growth is accompanied by a change in surface texture. The initial smooth, marble-like machined finish becomes rough and more clearly crystalline. On the basis of these observations, a mechanism was proposed to account for growth in polycrystalline TNT, involving the generation of shearing stresses at grain boundaries as a result of anisotropic thermal expansion, and relief of those stresses above a critical temperature (the melting point of the eutectic mixture) by a mass transfer process such as dynamic recrystallisation or grain boundary migration [6]. It was shown that although growth occurs equally in all directions in the randomised polycrystalline array of creamed TNT, in an ordered array growth depends on orientation within the crystalline matrix. These results are consistent with the growth mechanism proposed above [7].

This report examines further the dependence of the growth of TNT on the type and quantity of impurities present. In particular, the effects of the major impurities 2,4-dinitrotoluene and 2,3,4-trinitrotoluene are examined, as well as the influence of waxes commonly added as desensitisers for Composition B. Finally, the addition of anthracene and 1-nitronaphthalene, additives recommended to reduce exudation and/or cracking in commercial TNT charges [8], is also considered. The results of these experiments are discussed in terms of the mechanism proposed for the growth of TNT.

2. EXPERIMENTAL AND RESULTS

Creamed TNT (60 g) was prepared from pure TNT (purified by an extension of the industrial bisulphite process [9], followed by recrystallisation twice from ethanol), doped with 0-0.25% 2,4-dinitrotoluene (2,4-DNT) and cast into 20 mm diameter finger moulds. After cooling and solidification these castings were machined to right cylinders 50.8 mm long and 12.7 mm in diameter by means of a remote controlled lathe. The TNT cylinders were placed individually in test tubes, suspended in sets of six in a temperature controlled bath, and subjected to 40 six hour cycles 0-56°C. The axial growth after cycling was measured. The TNT cylinders were then subjected to 40 six hour cycles 10-66°C and total axial growth was measured. The sequences were repeated with cylinders of creamed TNT containing 0-0.25% 2,3,4-trinitrotoluene (2,3,4-TNT).

Typically, commercial TNT contains 0.10-0.40% 2,4-DNT and 0.03-0.10% 2,3,4-TNT, with other dinitro- and trinitrotoluenes being present in smaller quantities. Cylinders were prepared in the same fashion from pure TNT doped with 0.03% 2,3,4-TNT and 0-0.25% 2,4-DNT, and their axial growth was determined after 40 six hour cycles 0-54°C, and then after 40 six hour cycles 10-66°C. The sequence was repeated for cylinders of pure TNT containing 0.15% 2,4-DNT and 0-0.10% 2,3,4-TNT.

The results of these experiments are summarised in Table 1 below.

TABLE 1

GROWTH ON THERMAL CYCLING OF CREAMED PURE TNT DOPED WITH
2,4-DINITROTOLUENE AND 2,3,4-TRINITROTOLUENE

SERIES	2,4-DNT (%)	2,3,4-TNT (%)	GROWTH ON CYCLING 0-56°C (%)	GROWTH ON CYCLING 0-56°C & 10-66°C (%)	GROWTH ON CYCLING 0-56°C, 10-66°C & 15-71°C (%)
1	0	0	0	0	
	0.02	0	0.32	0.62	
	0.04	0	0.60	1.18	
	0.05	0	0.73	1.33	
	0.06	0	0.63	1.25	
	0.08	0	0.75	1.28	
	0.10	0	0.80	1.45	
	0.15	0	0.73	1.45	
	0.20	0	0.90	1.43	
	0.25	0	0.75	1.40	
2	0	0	0	0	0.08
	0	0.01	0	0	0.10
	0	0.02	0	0	0.22
	0	0.03	0	0	0.28
	0	0.04	0	0	0.30
	0	0.05	0.02	0.20	0.35
	0	0.10	0.03	0.25	0.45
	0	0.15	0.12	0.52	0.70
	0	0.20	0.05	0.44	0.68
	0	0.25	0.10	0.58	0.92
3	0	0.03	0	0	
	0.05	0.03	0.62	1.00	
	0.10	0.03	0.72	1.40	
	0.15	0.03	0.80	1.55	
	0.20	0.03	0.80	1.65	
	0.25	0.03	0.92	1.85	
4	0.15	0	0.70	1.35	
	0.15	0.02	0.70	1.48	
	0.15	0.04	0.83	1.58	
	0.15	0.06	0.97	1.80	
	0.15	0.08	0.93	1.78	
	0.15	0.10	0.83	1.48	

The effect of other impurities on growth of TNT was determined by preparing cylinders from creamed TNT containing 0.1% and 0.5% of paraffin wax, beeswax or the liquid dioctyl phthalate. To improve the dispersion of these additives in the TNT, similar charges were prepared with the addition of 0.5% n-octadecyl 2,4,6-trinitrobenzoate, which has been used as a surfactant for waxes in TNT [10]. The axial growth of these cylinders was measured after 40 six hour cycles 0-56°C, and again after 40 six hour cycles 10-66°C. The results of these experiments are presented in Table 2 below.

TABLE 2

GROWTH ON THERMAL CYCLING OF CREAMED PURE TNT DOPED
WITH NON-AROMATIC IMPURITIES

SERIES	ADDITIVE	GROWTH ON CYCLING 0-56°C (%)	GROWTH ON CYCLING 0-56°C & 10-66°C (%)
5	Pure TNT		
	0	0	0.10
	0.1% Paraffin Wax	0	0.10
	0.5% Paraffin Wax	0	0.13
	0.1% Beeswax	0	0.15
	0.5% Beeswax	0.03	0.20
	0.1% Dioctyl phthalate	0.13	0.33
	0.5% Dioctyl phthalate	0.28	0.58
6	Pure TNT + 0.5% <u>n</u> -octadecyl 2,4,6-trinitrobenzoate		
	0	0	0.03
	0.1% Paraffin Wax	-0.03	-0.03
	0.5% Paraffin Wax	-0.03	0.03
	0.1% Beeswax	0.03	0.08
	0.5% Beeswax	0.03	0.08
	0.1% Dioctyl Phthalate	0.05	0.10
	0.5% Dioctyl Phthalate	0.15	0.25

Finally, cylinders were prepared in the same manner from creamed commercial TNT (containing 0.11% 2,4-DNT, 0.01% 2,6-DNT, 0.03% 2,3,4-TNT and 0.04% 2,4,5-TNT) together with 0.25%, 0.50% and 1.00% anthracene and 1-nitronaphthalene. The axial growth of these samples was measured after 40 six hour cycles 0-60°C. The results are presented in Table 3 below.

TABLE 3

GROWTH ON THERMAL CYCLING OF CREAMED CRUDE TNT AND THE EFFECT OF
ANTHRACENE AND 1-NITRONAPHTHALENE

SERIES	ADDITIVE	GROWTH ON CYCLING 0-60° (%)
7	Crude TNT	1.15
	0.25% Anthracene	1.33
	0.5 % Anthracene	1.38
	1.0 % Anthracene	1.48
	0.25% 1-Nitronaphthalene	1.55
	0.5 % 1-Nitronaphthalene	1.65
	1.0 % 1-Nitronaphthalene	1.90

3. DISCUSSION

On the basis of our initial investigations into the growth of creamed TNT on thermal cycling, a model was proposed to account for the phenomenon, based on the ratchet mechanism of Burke and Turkalo for zinc [11], and illustrated graphically in Figure 1 [6]. This model considers a bicrystal in which the coefficient of expansion of grain 1 parallel to the grain boundary is greater than that of grain 2, and considers the stresses generated at that grain boundary as a consequence of thermal cycling. As the temperature is increased grain 1 attempts to expand more rapidly than grain 2, setting up a shearing stress across the grain boundary. This shearing stress increases until the elastic limit is reached, whereafter additional stress is accommodated by intracrystalline slip. The stress then remains constant until a critical temperature T^* , the eutectic melting point, above which a mass transfer process such as grain boundary sliding or dynamic recrystallisation allows relaxation of the shearing stress. Further temperature increase causes independent expansion of the individual grains, unimpeded by the grain boundary. The differential contraction of the grains as the bicrystal is cooled is also accommodated by the mass transfer process, until movement becomes impossible again below the critical temperature. The shearing stress is then set up in the opposite sense with grain 1 attempting to contract more rapidly than grain 2, and when the elastic limit is reached additional stress is again relieved by intracrystalline slip. When the temperature increases again during the next cycle the stresses decrease, pass through zero and then increase to the elastic limit [6].

Such shearing stresses must depend on the orientation of the TNT crystals both to their common boundary and one to another, and if they are to provide the driving force for growth of polycrystalline TNT then the phenomenon should show a dependence on orientation within the crystalline matrix. Three-dimensional growth appears to be independent of crystal orientation, but the extent of growth in a particular direction depends markedly on the orientation of the TNT crystals. Creamed TNT, with its random crystal orientation, grows equally in all directions. Growth is larger in a particular direction if there is a 'texture' of crystals with high and low coefficients of thermal expansion in that direction, which generate large intercrystalline stresses on thermal cycling. Growth is smaller when the crystals are so arranged that there are fewer such interactions and the intercrystalline stresses generated in that direction are smaller. These results are fully consistent with the 'ratchet' mechanism described above [7].

The presence of impurities in the TNT, and their nature and quantity should have a marked effect on the mass transfer process by which the intercrystalline stresses are relieved. Impurities which are present in a liquid are not evenly dispersed throughout the polycrystalline assembly on solidification, but rather tend to concentrate at the grain boundaries. Thus the low-melting eutectic mixtures in polycrystalline commercial TNT are concentrated where the stresses are generated during thermal cycling. The relief of those stresses might be expected to depend on both the quantity of liquid and the period of the time the liquid is present at the grain boundary, determined by the melting point of the eutectic mixture. These factors are determined by the quantity and nature of the impurities present.

The individual effects on the growth of creamed TNT of added 2,4-DNT and 2,3,4-TNT are illustrated in Figures 2 and 3. Figure 2 indicates that the effect of 2,4-DNT on the growth of creamed TNT is a step-function; growth increases markedly with 2,4-DNT concentrations up to about 0.10%, whereafter the addition of further impurity appears to have little effect. This may be evidence that the mass transfer process is grain boundary sliding, which would depend on the presence of a quantity of liquid between the crystals, rather than dynamic recrystallisation which would depend on the quantity of liquid present. The effect of 2,3,4-TNT is much less than that of 2,4-DNT, with no significant growth being observed on cycling 0-56°C and small but significant growth on cycling 10-66°C. Only after recycling 15-71°C was growth in excess of 0.7% observed. It should be noted that 2,4,6-TNT forms binary eutectics of melting point 46.3°C and 67.3°C respectively with 2,4-DNT and 2,3,4-TNT [1]. Significant growth was not observed until these temperatures were approached but it was detected slightly below these temperatures. This may be due to the presence of undetected impurities which further lowered the eutectic melting point, but grain boundary sliding is frequently observed before the first liquid is detected. This may then be further support for grain boundary sliding as the mass transfer process. It should also be noted that a plateau in the growth/impurity relationship was not detected below 0.25% 2,3,4-TNT.

The presence of 2,3,4-TNT alone appears to have comparatively little influence on the growth of creamed TNT on thermal cycling. However the presence of this material significantly lowers the melting point of mixtures of 2,4,6-TNT and 2,4-DNT [1], and experiments were carried out to ascertain the effect of 2,4-DNT and 2,3,4-TNT in concert on the growth of TNT at about

the concentrations at which they tend to occur in the commercial explosive. Figure 4 shows the growth of TNT containing 0.03% 2,3,4-TNT and up to 0.25% 2,4-DNT, and comparison with Figures 2 and 3 demonstrates that growth is not a simple summation of the contributions of the 2,4-DNT and 2,3,4-TNT, but rather there is a synergistic effect of the two impurities. Similarly Figure 5 shows the growth of TNT containing 0.15% 2,4-DNT and up to 0-0.10% 2,3,4-TNT. Again growth is clearly not simply a summation of the contributions of the component impurities, but rather first increases with the concentration of 2,3,4-TNT, reaching a maximum at about 0.07% of that component, and then decreases. The ternary eutectic of 2,4,6-TNT with 2,4-DNT and 2,3,4-TNT melts at 37°C and contains 42.6% 2,4-DNT and 18.85% 2,3,4-DNT by weight, a weight ratio of 2.26 : 1 [1]. This is within experimental error of the ratio of impurities at which maximum growth occurs.

The effect on the growth of creamed TNT on thermal cycling of nitroaromatic impurities which form low-melting eutectics with the TNT is amply illustrated above. The effect of other impurities which do not form such eutectics (waxes which are added to TNT-based explosives as phlegmatisers or desensitisers) is illustrated in Table 2. The presence of 0.1% or 0.5% of paraffin wax (mp 61.2°C) or beeswax (mp 61-4°C) caused no detectable growth in TNT cycled 0-56°C, and insignificant growth on cycling 10-66°C. On the other hand the presence of 0.1% or 0.5% dioctyl phthalate enhanced growth for either cycling regime. This may be due to the fact that dioctyl phthalate is a liquid, or it may be a reflection of the significant solubility of TNT in dioctyl phthalate. Further, the inclusion of n-octadecyl 2,4,6-trinitrobenzoate (added to improve the dispersion of the additives) does nothing to enhance growth on thermal cycling. Indeed there is a marginal reduction in growth in each case.

Finally the effect of added anthracene and 1-nitronaphthalene on the growth of commercial TNT is detailed in Table 3. These materials have been recommended as additives to reduce exudation and/or cracking in commercial TNT charges. Table 3 demonstrates that these additives increase rather than decrease the growth of TNT on thermal cycling, with the effect of 1-nitronaphthalene being greater than that of anthracene. However, it was noticed that exudation from the charges was also enhanced rather than suppressed by the presence of these additives, the effect again being greater for 1-nitronaphthalene. The usefulness of these materials as additives for TNT-based charges is therefore brought into question.

In the initial formulation of the 'ratchet' mechanism to account for growth of polycrystalline TNT on thermal cycling [6] it was proposed that the mass transfer process by which intercrystalline stresses were relieved might be grain boundary sliding or a form of dynamic recrystallisation. No evidence was obtained for grain boundary sliding, but photomicrography of a machined surface of creamed TNT, polished and etched with bromoform, indicated a thermal etching of the surface accompanied by a degree of recrystallisation. The surface initially consisted of an array of crystals with distinct, straight line edges and angular corners. On cycling the grain boundaries became more diffuse and the sharp corners were smoothed as though by erosion. Simultaneously, smaller regular crystals (cubes and prisms) appeared and then grew as thermal cycling was continued. It was recognised, of course, that processes occurring in the bulk of the polycrystalline mass may not occur on the surface, and the converse may also be true.

The explanation proposed [6] was that impurities (including 2,4-dinitrotoluene and 2,3,4-trinitrotoluene) accumulate at the grain boundaries, leaving the individual TNT crystals relatively free of impurities. As the temperature increases the eutectic mixture at the grain boundaries melts preferentially, broadening the boundaries and 'softening' the edges and corners. On cooling the TNT resolidifies as cubes and prisms. On the basis of these observations, however, the possibility that these phenomena may result simply from exposure to the higher temperatures could not be excluded. Indeed a sample of TNT held at 56°C for 16 days, under which conditions no growth took place, suffered more thermal etching and recrystallisation than did an identical sample subjected to 6 hour thermal cycles 0-56°C for 32 days. It therefore seems certain that the thermal etching and recrystallisation result from exposure to elevated temperatures rather than from thermal cycling to them. There is, then, less evidence than previously believed for the proposal that dynamic recrystallisation is the mass transfer process involved, and the effects of impurities described in this report perhaps agree better with grain boundary sliding being that mass transfer process.

4. CONCLUSIONS

The growth on thermal cycling of polycrystalline TNT increases with the concentrations of 2,4-dinitrotoluene and 2,3,4-trinitrotoluene individually (these materials are major impurities in commercial TNT), but these materials have a synergistic effect on growth when they are present together. On the other hand paraffin wax and beeswax, commonly added as desensitisers to TNT-based compositions, have no effect on the growth process. Finally anthracene and 1-nitronaphthalene increase growth in polycrystalline TNT. These results are fully in accord with the mechanism proposed previously to account for the growth process.

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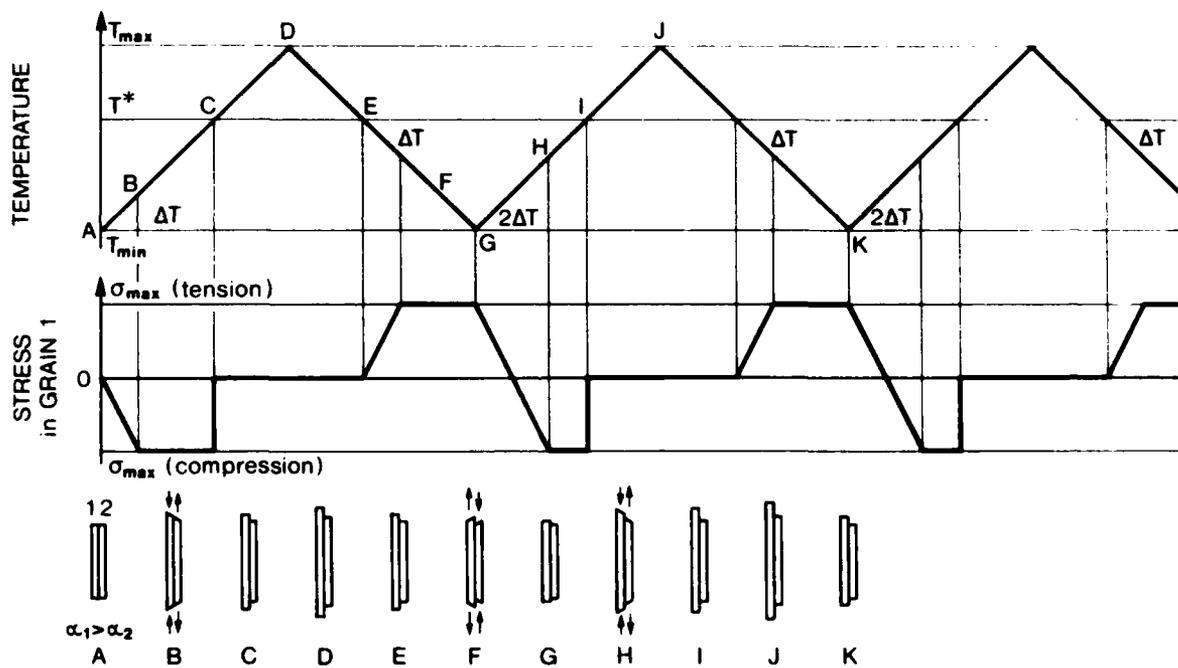


FIGURE 1 - Burke and Turkalo's model for growth in a zinc bicrystal.

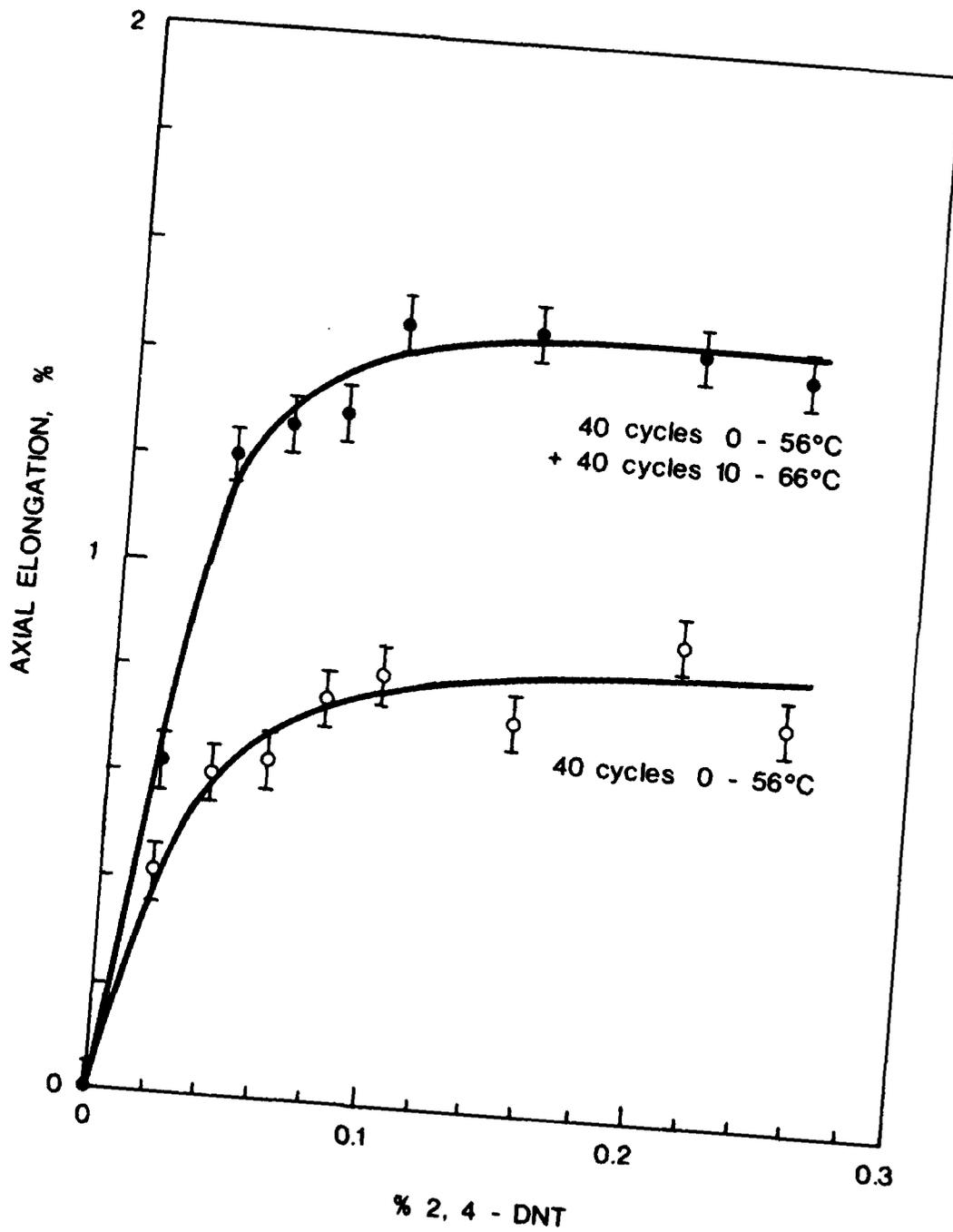


FIGURE 2 - Growth of TNT containing 2,4-DNT.

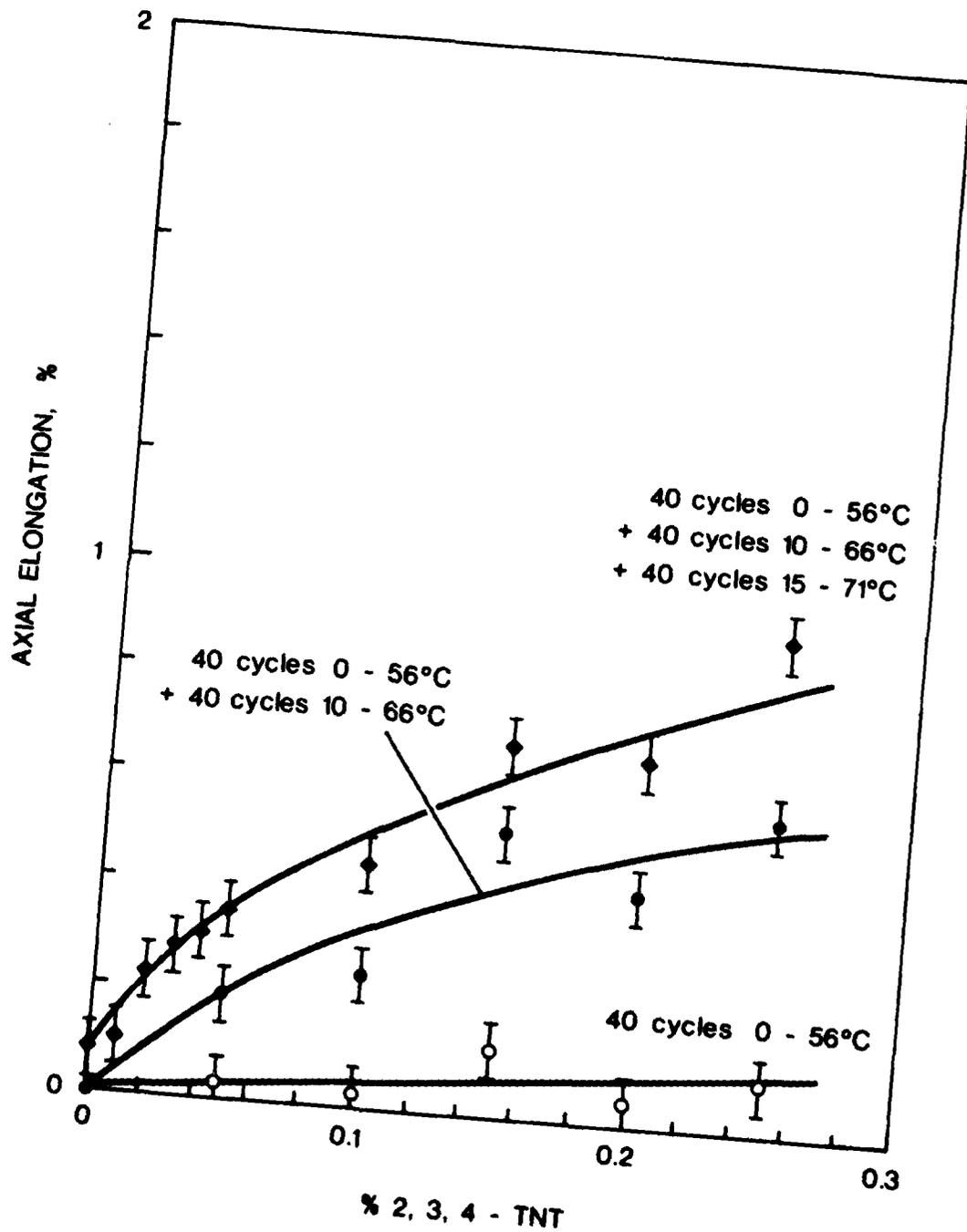


FIGURE 3 - Growth of TNT containing 2,3,4-TNT.

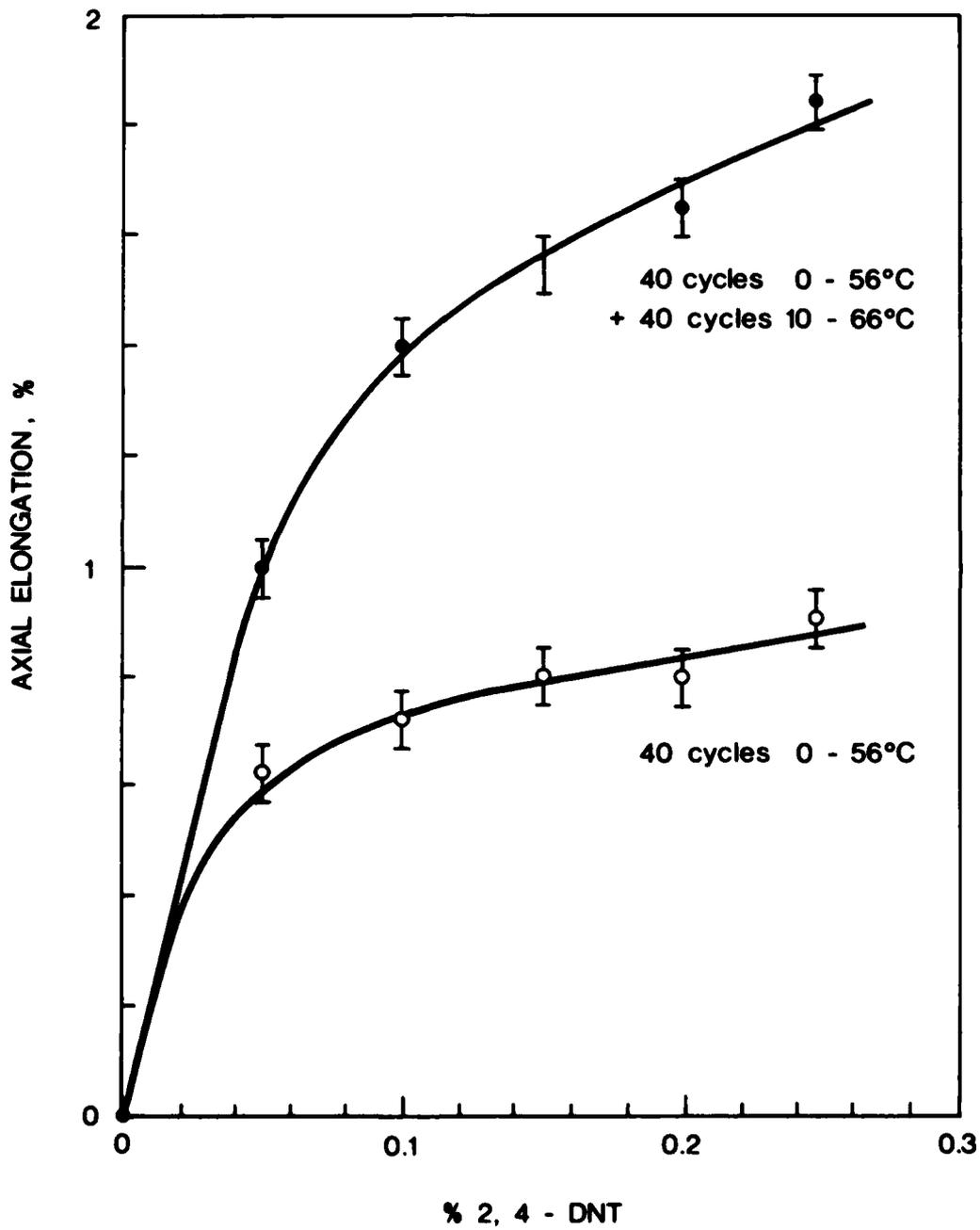


FIGURE 4 - Growth of TNT containing 2,4-DNT and 0.03% 2,3,4-TNT.

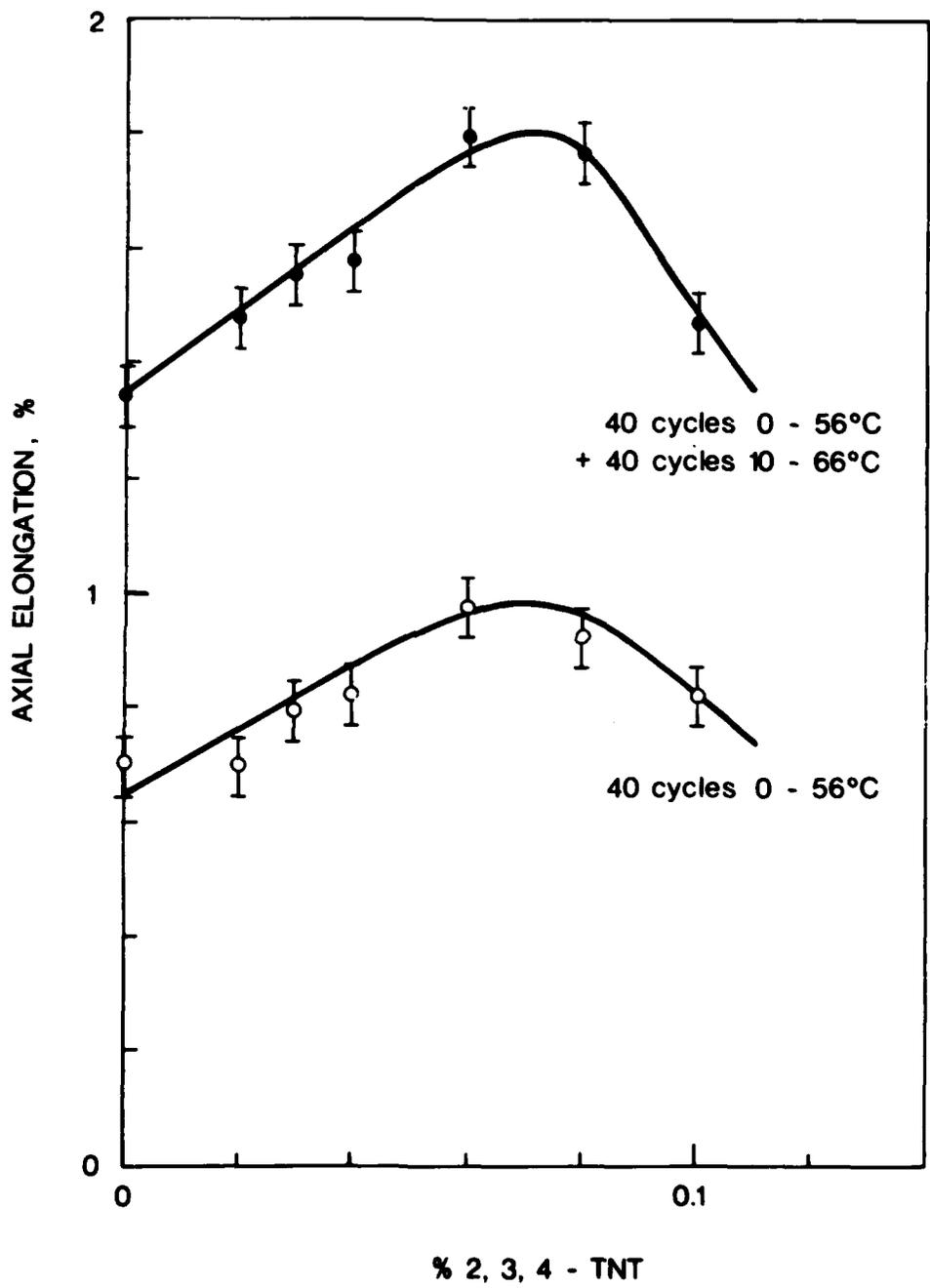


FIGURE 5 - Growth of TNT containing 2,3,4-TNT and 0.15% 2,4-DNT.

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