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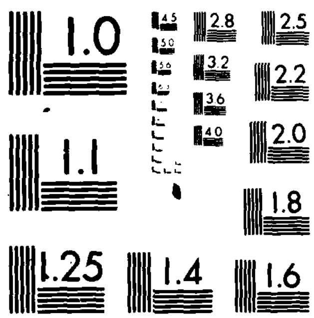

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INTERCALATED COMPOUNDS: A NEW CLASS OF MATERIALS AS ADVANCED SOLID LUBRICANTS

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25 January 1982

PROGRESS REPORT
TASK AREA NO. Z02208
Work Unit No. GC-125

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The results presented in this report demonstrate that the process of intercalation, i.e., the formation of chemical compounds via insertion of atomic or molecular species in the van der Waals gap between planes of lamellar solids can substantially improve the intrinsic lubricating properties of solids. Using graphite as a model host compound, various transition metals and metal chlorides intercalated into graphite were formulated into solid film lubricants and their lubricating properties determined in a laboratory wear test

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered) device. Comparisons of endurance life and load carrying capacity are made relative to molybdenum disulfide and unintercalated graphite. Graphite/19.8 wt. % $CoCl_2$ was found to exhibit over a five-fold increase in endurance life while graphite/19.3 wt. % $NiCl_2$ provided a greater than two-fold increase in load carrying capacity relative to graphite and was equivalent to MoS_2. The degree of improvement in endurance life was found to be dependent on the concentration of intercalant in graphite and the resulting increase in interlayer carbon spacing due to intercalation. A total of 23 different intercalate compounds were investigated at various concentration levels.

SUMMARY

INTRODUCTION

Solid lubricants are generally defined as materials which produce lubrication on two relatively moving surfaces under essentially dry conditions as opposed to oil or grease lubrication. Molybdenum disulfide (MoS_2) and graphite are probably the best known lubricating solids, the former being used in the majority of applications because of its overall lubricating ability. The use of solids as lubricants encompasses a variety of techniques such as bonded solid films, burnished films, self-lubricated solid composites as well as additives to grease and oil or as a paste. Depending on the application, one form will be more desirable than the others. Solid lubrication is utilized throughout DoD Weapons Systems where relubrication is impractical or impossible, temperatures exist beyond which oils or greases are stable and where high loads are encountered. Indeed, in some applications, solids are the only means for providing adequate lubrication. The most common usage of solids as lubricants is in the form of a bonded films. Typical formulations consist of lubricating pigment (MoS_2) incorporating either organic (phenolic, epoxy, polyimide, etc.) or inorganic (silicate, phosphate, etc.) binders. Conventional paint application techniques such as dipping, brushing or spraying are used to deposit films on component surfaces. A film thickness on the order of 0.0005 inches have been found to be optimum. Thicker coatings wear rapidly as a result of cohesive forces. Thinner coatings result in only limited durability. At present coatings based on MoS_2 have been found to provide the best available lubricating film. The most obvious disadvantage of solid film lubricants is their limited durability. For the most part research efforts to enhance durability have centered on the study of alternate binders and surface pretreatments. Some work has been performed on improving lubricating pigments, but MoS_2 still ranks as the best material available today. This material is obtained from molybdenite ore which is also converted to molybdenum metal for use in the steel industry. A recent trend toward increased production of molybdenum metal could lead to shortages of MoS_2 for lubricating purposes. With these deficiencies in mind, an independent research program was initiated at the Naval Air Development Center (NAVAIRDEVCEN) in October 1980 based on studying intercalated solids as advanced solid lubricants. The title of the work unit is "Intercalated Compounds: A New Class of Materials as Advanced Solid Lubricants", Task Area No. Z02208 Work Unit GC-125.

RESULTS

1. Transition metals and metal chlorides intercalated into the van der Waals gap between layers of carbon atoms in graphite can substantially improve its lubricating properties relative to molybdenum disulfide (MoS_2) and unintercalated graphite.

2. The endurance life of solid film lubricants containing intercalated graphite compounds can be improved by as much as seven-fold compared to MoS_2 and unintercalated graphite.

3. For transition metal chlorides intercalated into graphite the degree of improvement was found to increase linearly with increasing molar concentration of intercalant and appears to be independent of the chemical nature of the transition metal chloride.

4. Graphite/transition metal intercalates which possess a 40% smaller interplanar distance than the transition metal chlorides gave lower increases in endurance life on a molar concentration basis compared to the metal chlorides.

5. The load carrying capacity of a graphite/20% $NiCl_2$ intercalate was found to be 2.2-fold greater than graphite and equal to MoS_2.

CONCLUSION

The process of intercalation has been shown to provide enhanced solid lubricant performance using graphite as a model host compound. The extension of this phenomenon to other lamellar compounds may provide a basis for synthesis of "tailor-made" solid lubricants as well as provide insight into the fundamental mechanisms governing the action of lubricating solids in general.

FUTURE PLANS

As with any new concept, the initial investigation usually raises more questions about the nature of the effect than are answered. The following short term studies are planned in order to better understand this phenomenon:

1. Investigate the effect of concentration of intercalant on lubricating properties for a given intercalant.

2. Determine the effect of other classes of compounds such as transition metal sulfides and oxides which are known to intercalate into graphite.

3. Study alternate host materials such as boron nitride.

4. Investigate the feasibility of co-intercalation to uncover possible synergistic effects.

Long range plans include optimizing synthesis techniques, characterization of synthesized compounds by x-ray analysis, and the determination of chemical changes occurring on metallic surfaces lubricated with intercalated solids.

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BACKGROUND

The ability of graphite to form compounds by intercalation, i.e., the formation of chemical compounds by insertion of atomic or molecular species in the van der Waals gap between carbon layers has been known since 1859 (1). Since that time, a variety of intercalated compounds have been prepared predominately with transition metal chlorides (2). Several excellent reviews on the subject of intercalation compounds appeared in 1959 and 1960 (3), (4), (5), (6) and a resurgence of interest in the unique properties of these materials in the 1970's (7) led to the first international conference to be held on graphite compounds (8) and the introduction of a new journal titled "Synthetic Metals", integrating research and applications on intercalation compounds of graphite, transition metal compounds, and quasi one-dimensional conductors.

Three classes of graphite intercalation compounds have been described, namely:

1. Non-conducting or covalent: Graphite oxide and graphite fluoride are the only known examples. In these compounds, the classic properties of graphite are lost, i.e., the planar structure of the carbon atoms becomes buckled and the materials become non-conductive.

2. Conducting or ionic: Most compounds fall into this class which can be further distinguished by whether the intercalant gives up or receives electrons. For example, alkali metal intercalants are designated donor compounds while metal chloride intercalants are acceptor compounds.

3. Residue compounds: There is some question as to whether these materials are true compounds since the intercalant is retained only at imperfections and peripheral surfaces of the graphite crystal, after decomposition or reduction has taken place.

Since most of the compounds explored in this study fall under the ionic classification, a few additional remarks about them are in order. When compounds of this type are analyzed by x-ray diffraction it is found that not all of the layers of the crystal are necessarily intercalated. This observation has led to the use of the term "stage" to describe the extent of intercalation. As shown in Figure 1, a Stage 1 compound is one in which intercalant can be found between every layer of carbon atoms. In Stage 2 compounds, every other layer of carbon atoms is intercalated and so on for Stage 3 and above. No lamellar compound has been found to contain multiple layers of intercalant between adjacent carbon planes (4). As a consequence of intercalation, the interplanar distance between intercalated carbon layers is increased by as much as three-fold. This increased separation should lead to a weakening of the van der Waals forces (i.e., lowering of interplanar binding energy) between planes thus allowing slippage of one plane of atoms over the other more readily. The result of this phenomenon should therefore translate into more effective solid lubrication. It is known that effective lubrication with graphite is dependent on minute quantities of vapor (O_2, H_2O, etc.) supporting the crystal structure of carbon atoms (9). In vacuum and at elevated temperatures in an inert atmosphere failure of graphite to lubricate has been attributed to collapse of carbon layers as a result of depletion of these vapors. Thus, graphite is not an intrinsic lubricant but may be caused to lubricate under certain conditions.

Recent proposals have been made regarding the use of intercalated graphite compounds as organic reagents (10), catalysts (11), battery cathodes (12) and highly conducting materials (13). The obvious application which appears to have been overlooked is as improved lubricating solids. Only one compound, namely; graphite fluoride, one of the non-conducting types, has been thoroughly investigated by the U. S. Army (14), NASA (15) and the U. S. Air Force (16) with the conclusion that while graphite fluoride looked promising as a solid lubricant in applications involving low stress, it was not considered suitable for heavy load requirements. Since this material is not representative of the majority of intercalated graphite compounds, a study was undertaken on a variety of graphite intercalation compounds in order to determine lubricant performance under high loads.

E X P E R I M E N T A L

Intercalated graphite compounds were obtained from a commercial source. The powders were ground in a mortar and pestal and only that portion which passed through a 150 micron sieve used. Apparent densities were determined by a procedure similar to that described in reference (17). In addition to the use of commercially available graphite/ferric chloride, synthesis of this material was also undertaken. Graphite (44 micron, used as received) and anhydrous ferric chloride (used as received) were thoroughly mixed in a 1 to 3 ratio respectively and sealed in carius tubes. The tubes were heated at 316°C (600°F) for 24 hours. After cooling to room temperature, the tubes were broken and the recovered powder weighed. This powder was washed with copious quantities of distilled water to remove unreacted ferric chloride followed by washing with acetone. After drying at 66°C (150°F) the powder was then weighed. Knowing the initial concentration of graphite and the differences in weight between the unwashed and washed powder, an estimate of the degree of intercalation of ferric chloride into graphite was calculated. In order to verify the presence of intercalated ferric chloride a small sample of the powder (50 mg) was placed in a crucible and heated at 649°C (1200°F) for 30 minutes. The presence of intercalated ferric chloride was evident from the red deposits of Fe_2O_3 formed. This deposit was not found with samples of initial reaction mixture which had been washed free of ferric chloride. Thus, the red Fe_2O_3 formed was from the decomposition of ferric chloride which had been intercalated in the graphite structure.

These powders were formulated into solid film lubricants and investigated as a lubricating source on the falex lubricant tester. A simple formulation using only lubricating solids and MIL-R-3043 phenolic resin as a binder was employed. A formulation based on 33 wt. % MoS_2 (MIL-M-7866) and 67 wt. % MIL-R-3043 (34 wt. % solids) was used as a standard. All other formulations were prepared using the same volume of lubricating solids as MoS_2. This was calculated using the apparent density. The lubricating film was formed by spraying the solid lubricant formulation on the test specimens providing a coating with a film thickness in the range of 0.005-0.0127 mm (0.0002-0.0005 inches). The test specimens were then heated at 149°C (300°F) for one hour.

The Falex lubricant tester which was used to study the lubricating properties of the resultant films has been previously described (18). Essentially, a 6.35 mm (.25 in.) diameter cylindrical pin (AISI-3135 steel) is rotated at 290 RPM against two stationary V-blocks (AISI-1137 steel) under load. Two properties of the solid film lubricant were determined, i.e., endurance life and load carrying capacity (ASTM D 2625). Surface preparation of the test specimens consisted of grit blasting with 120 steel grit which produced a surface roughness of 50-60 RMS. The results reported are average of at least four determinations.

R E S U L T S

A preliminary investigation centered on the study of graphite/ferric chloride since other properties of this material have been comprehensively studied (19), (20), (21), (22). Table 1 shows the results of endurance life and load carrying capacity determinations on solid film lubricants containing MoS_2, graphite, graphite/ferric chloride intercalate and a graphite-ferric chloride physical mixture. A dramatic increase in the endurance life (>2.7 fold) of the intercalated graphite compound was observed compared to MoS_2 and normal graphite. The load carrying capacity also increased relative to graphite. In order to demonstrate that it is the process of intercalation that is playing a key role in achieving increased endurance life and load carrying capacity, a physical mixture of graphite containing 8.2 wt. % ferric chloride was also investigated. The physical mixture did exhibit an improvement in endurance life relative to graphite (1.8 fold), however, it was not as pronounced as for the graphite/ferric chloride intercalation compound (3.7 fold). No improvement was observed in load carrying capacity.

Having demonstrated the potential for the process of intercalation, a series of compounds were investigated in order to determine if the effect could be achieved with a wider range of materials. The results of this study are presented in Tables 2 (Endurance Life) and 3 (Load Carrying Capacity). The compounds studied were divided into three classes, namely; graphite/metal chlorides, graphite/metals and graphite/miscellaneous intercalates. For endurance life (Table 2), the average value of at least four tests is presented in addition to an endurance life ratio which is defined as the average endurance life of the graphite/intercalate compound divide by the average endurance life of normal graphite. This is presented to show the degree of improvement or lack of improvement relative to graphite. Of the eleven graphite/metal chloride intercalates studied (mainly, transition metal chlorides) four compounds, namely; 19.8 wt. % $CoCl_2$, 13.7 wt. % $CuCl_2$, 8.2 wt. % $FeCl_3$ and 5.0 wt. % YCl_3 exhibited a longer endurance life than graphite by factors of 7.6, 5.1, 3.7 and 2.3 respectively. The balance of the compounds gave a lower endurance life relative to graphite. Eight graphite/transition metal intercalates were studied, three of which exhibited improvements ranging from 3.2 (10.0 wt.

% Co), 3.2 (4.0 wt. % Fe) to 1.5 (5.1 wt. % Cu). No improvement was obtained with graphite oxide, 47.8 wt. % SbF_5, graphite fluoride and 58.2 wt. % CrO_3.

Table 3 shows the results of load carrying capacity determinations on the same series of compounds. Only one compound (19.3 wt. % $NiCl_2$) was found to equal MoS_2 and exhibited a 2.2 fold increase in load carrying capacity relative to graphite. Other graphite/metal chloride intercalates exhibiting improved load carrying capacity over graphite included 19.8 wt. % $CoCl_2$ (1.8), 8.2 wt. % $FeCl_3$ (1.6) and 13.7 wt. % $CuCl_2$ (1.3). The balance of the compounds were either equal to or less than graphite. Of the graphite/metal intercalates 10.0 wt. % Co, 14.0 wt. % Ni and 4.0 wt. % Fe showed improvements of 2.0, 2.0 and 1.5 respectively. For the miscellaneous compounds, graphite oxide was found to provide a 1.5 fold improvement and 47.8 wt. % SbF_5 1.2 fold. Graphite fluoride was found to be equal to graphite.

Synthesis of a graphite/ferric chloride intercalate performed in this laboratory yielded a compound containing approximately 5 wt. % $FeCl_3$. An average endurance life of 105 minutes (2.1 fold increase) and a load carrying capacity of 10,008 N (2250 lbs.) (1.5 fold increase) was found for this compound.

DISCUSSION

The data presented in this paper were generated during the initial phase of an investigation to determine whether beneficial effects on solid lubrication could be achieved via the process of intercalation. All of the graphite intercalate compounds listed in Tables 1, 2 and 3 were obtained as catalogue items from a commercial source with only one concentration range available for each compound, thus, a concentration range of 1.2 to 58.2 wt. % intercalant is represented. Some compounds were not studied due to their known extreme reactivity with air and moisture sensitivity such as the graphite/alkali metal intercalates.

If the mole percent concentration of intercalant is plotted versus the average endurance life for those graphite/metal chloride compounds with an endurance life ratio of >1, esstentially a linear relationship is obtained as shown in Figure 2 and represented by equation (1).

$$L_{MC} = 165 X + 51 \qquad (1)$$

where:

L_{MC} = average endurance life in minutes of graphite/metal chloride intercalate

X = mole percent intercalant in graphite

The linear dependence of wear life on concentration appears to indicate that the various increases in endurance life observed are dependent on the molar quantity of intercalant present rather than the chemical nature of the transition metal chloride. This result would be expected if the predominant effect on lubrication depends on the weakening of interlayer binding forces. As shown in Table 4, the distance separating intercalated carbon layers is approximately the same for metal chloride intercalants thus the decrease in binding energy should also be equal. The distance in normal graphite is 0.335 nm.

In regard to graphite/transition metal intercalates, their interplanar separation is approximately 40% lower than the metal chlorides (see Table 4) thus binding energies for these materials would be higher and the resultant increase in wear life over graphite is expected to be lower than with the chlorides. On a mole concentration basis this was indeed observed for those materials with an endurance life ratio >1. (See Figure 2). A fairly linear relationship as represented by equation (2)

$$L_M = 43\,X + 51 \qquad (2)$$

where:

L_M = average endurance life in minutes of graphite/metal intercalate

was found for 10.0 wt. % Co and 5.1 wt. % Cu intercalants, however, the 4.0 wt. % Fe compound exhibited a higher endurance life than would be predicted from equation (2). The reason for this anomalous behavior is not understood at this time.

The effect of interplanar separation on endurance life can be further demonstrated as shown in Figure 3. Using equations (1) and (2), L_{MC} and L_M were calculated for X = 2.0. These values were plotted as a function of interplanar distance between carbon layers for the particular class of intercalant. Also included is normal graphite. The endurance life can be observed to increase linearly with increasing carbon layer separation.

It is interesting to note that in general if a particular graphite compound was better than graphite in endurance life, it was also better in load carrying capacity, and that if it was equal to or poorer than graphite the same held true. The exceptions to this observation are 5.0 wt. % YCl_3 and 5.1 wt. % Cu which had better wear life than graphite but equal to or poorer load carrying capacity. On the other hand, 19.3 wt. % $NiCl_2$, graphite oxide and 47.8 wt. % SbF_5 had a poorer wear life but better load carrying capacity. The load carrying capacity of the 19.3 wt. % $NiCl_2$ was found to be equal to that of MoS_2.

In regard to the mechanistic considerations involved in achieving enhanced lubrication with intercalated compounds, a simplified explanation based on the increase in separation of graphite layers was initially invoked. It is well known that insertion of atoms or molecules in the van der Waals gap of layered compounds causes an expansion of the lattice. This expansion results in a weakening of the van der Waals forces between planes thus allowing for easier shear and better lubrication. Jamison (23) has shown that the lubricating properties of transition metal dichalcogenides (TMD's) are determined by the number of electrons in the ψ_7 nonbonding band. When the electrons in this band have a wavelength which satisfies the Bragg condition, they are reflected from the {002} planes thus exchanging momentum with the lattice and causing an effective pressure which expands the lattice. The resulting expansion in the direction perpendicular to the basal planes thus allows for easier shear as mentioned above and results in good lubricating properties. Molybdenum disulfide and other Group VI TMD's have the proper concentration of electrons in the ψ_7 band to provide good lubrication. Group V TMD's, which appear to be electron deficient and thus poor lubricants can be made into effective lubricants with the proper electron concentration by the formation of binary solid solutions (24). Jamison also indicated that the electron concentration could also be altered by intercalation and suggested that compounds of MoS_2 intercalated with alkali metals should lead to improved lubrication, however, no lubrication data was available for these compounds. It is considered that the same type of reasoning used to explain the lubricating properties of TMD's can be applied to graphite/intercalate compounds.

The above discussion centered on explaining the beneficial effects of intercalation yet a substantial amount of data was reported in which no improvement was obtained or extreme deterioration of the lubricating properties of graphite was observed. Several observations can be made regarding these results.

1. The concentration of intercalant is less than 5 wt. % where no effect is observed due to low concentrations.

2. The graphite/intercalate compound decomposes under the temperature and pressure of the wear test yielding free intercalant which disrupts lubricating action.

3. In these compounds lattice dimensions are decreased rather than increased thus strengthening the bonding between layers. There is no precedent in the literature for this effect although not all of these materials have been thoroughly characterized.

4. The poorer lubricating compounds with relatively high concentrations of intercalant, such as 37.0 wt. % $AlCl_3$ and 47.8 wt. % SbF_5 are inherently unstable. $AlCl_3$ can be hydrolyzed in the intercalate state (5) suggesting that bonding between the carbon planes with $AlCl_3$ is different than with the transition metal chlorides.

5. The compound is in reality a physically mixture and not intercalated.

Of course all of these observations are subject to further investigation.

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TABLE 1. FALEX TEST RESULTS

Solid Film Lubricant	Average Endurance Life (4448N (1000 Lb) Gage Load) (Minutes)	Load Carrying Capacity Newtons (N)	Load Carrying Capacity (lb)
MoS_2	68	14,457	(3250)
Graphite	51	6,672	(1500)
Graphite/8.2 wt. % $FeCl_3$ (Intercalate)	187	11,121	(2500)
Graphite-8.2 wt. % $FeCl_3$ (Physical Mixture)	90	6,672	(1500)

TABLE 2. FALEX ENDURANCE LIFE TEST RESULTS

Solid Film Lubricant	Average Endurance Life (4448N (1000 Lb) Gage Load) (Minutes)	Endurance Life Ratio (Graphite Intercalate to Graphite)
Graphite/Metal Chlorides		
19.8 wt. % $CoCl_2$	390	7.6
13.7 wt. % $CuCl_2$	258	5.1
8.2 wt. % $FeCl_3$	187	3.7
5.0 wt. % YCl_3	116	2.3
19.3 wt. % $NiCl_2$	45	0.88
4.3 wt. % $RuCl_3$	45	0.88
1.2 wt. % $PdCl_2$	35	0.69
7.3 wt. % $CrCl_3$	27	0.53
1.2 wt. % $PtCl_4$	15	0.29
1.5 wt. % $RhCl_3$	15	0.29
37.0 wt. % $AlCl_3$	7	0.14
Graphite/Metals		
10.0 wt. % Co	163	3.2
4.0 wt. % Fe	161	3.2
5.1 wt. % Cu	78	1.5
14.0 wt. % Ni	43	0.84
2.3 wt. % Rh	30	0.59
1.6 wt. % Ru	7	0.14
1.7 wt. % Pd	7	0.14
2.2 wt. % Pt	7	0.14
Graphite/Miscellaneous		
Oxide (62.2 wt. % C)	37	0.73
47.8 wt. % SbF_5	30	0.59
Fluoride $(CF_x)_n$ x >1	10	0.20
58.2 wt. % CrO_3	0	0

TABLE 3. FALEX LOAD CARRYING CAPACITY TEST RESULTS

Solid Film Lubricant	Load Carrying Capacity Newtons (N)	(Lbs)	Load Carrying Capacity Ratio (Graphite Intercalate to Graphite)
Graphite/Metal Chlorides			
19.3 wt. % $NiCl_2$	14,457	(3250)	2.2
19.8 wt. % $CoCl_2$	12,233	(2750)	1.8
8.2 wt. % $FeCl_3$	11,121	(2500)	1.6
13.7 wt. % $CuCl_2$	8,896	(2000)	1.3
7.3 wt. % $CrCl_3$	6,672	(1500)	1.0
5.0 wt. % YCl_3	6,672	(1500)	1.0
1.2 wt. % $PtCl_4$	6,672	(1500)	1.0
4.3 wt. % $RuCl_3$	6,672	(1500)	1.0
1.2 wt. % $PdCl_2$	5,560	(1250)	0.83
1.5 wt. % $RhCl_3$	5,560	(1250)	0.83
37.0 wt. % $AlCl_3$	4,448	(1000)	0.67
Graphite/Metals			
10.0 wt. % Co	13,344	(3000)	2.0
14.0 wt. % Ni	13,344	(3000)	2.0
4.0 wt. % Fe	10,008	(2250)	1.5
5.1 wt. % Cu	5,560	(1250)	0.83
2.3 wt. % Rh	5,560	(1250)	0.83
2.2 wt. % Pt	5,560	(1250)	0.83
1.7 wt. % Pd	4,488	(1000)	0.67
1.6 wt. % Ru	3,336	(750)	0.50
Graphite/Miscellaneous			
Oxide (62.2 wt. % C)	10,008	(2250)	1.5
47.8 wt. % SbF_5	7,784	(1750)	1.2
Fluoride $(CF_x)_n$ x >1	6,972	(1500)	1.0
58.2 wt. % CrO_3	1,334	(300)	0.2

TABLE 4. INTERPLANAR DISTANCE BETWEEN INTERCALATED CARBON LAYERS

Graphite/Metal Chlorides (Ref 7)	Interplanar Distance (nm)
$FeCl_3$	0.937
$CoCl_2$	0.950
$CuCl_2$	0.940
YCl_3	0.963
Graphite/Metals (Ref 25)	
Fe	0.565
Co	0.563
Cu	0.561

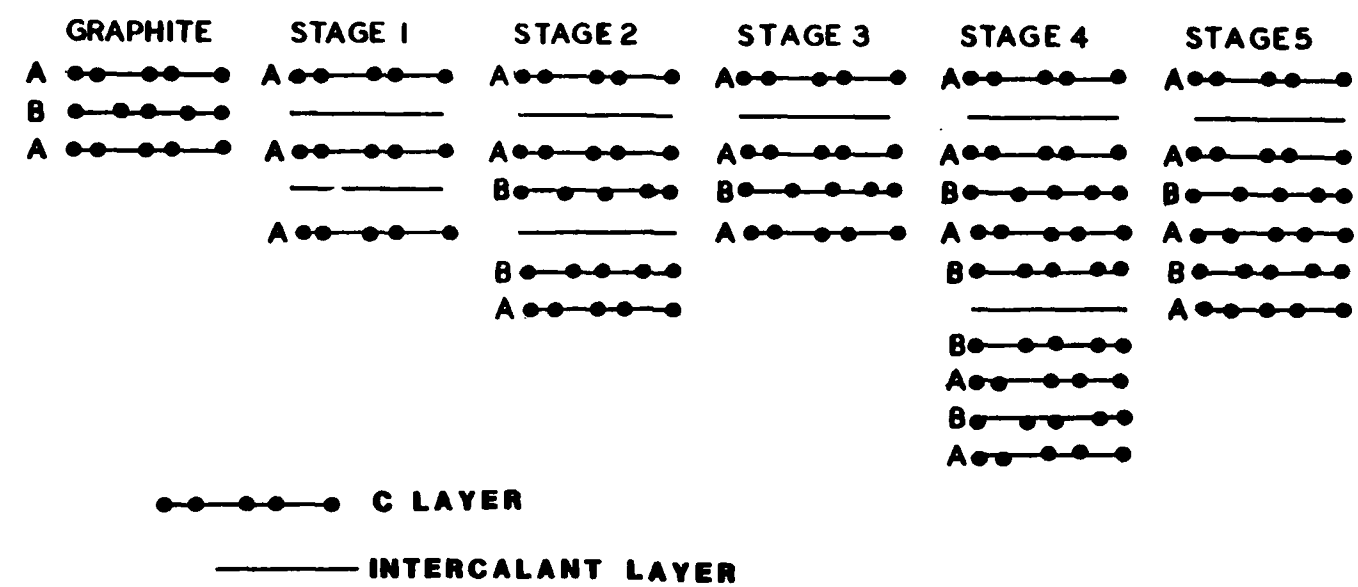

FIGURE 1 REPRESENTATION OF STAGES IN GRAPHITE INTERCALATION COMPOUNDS

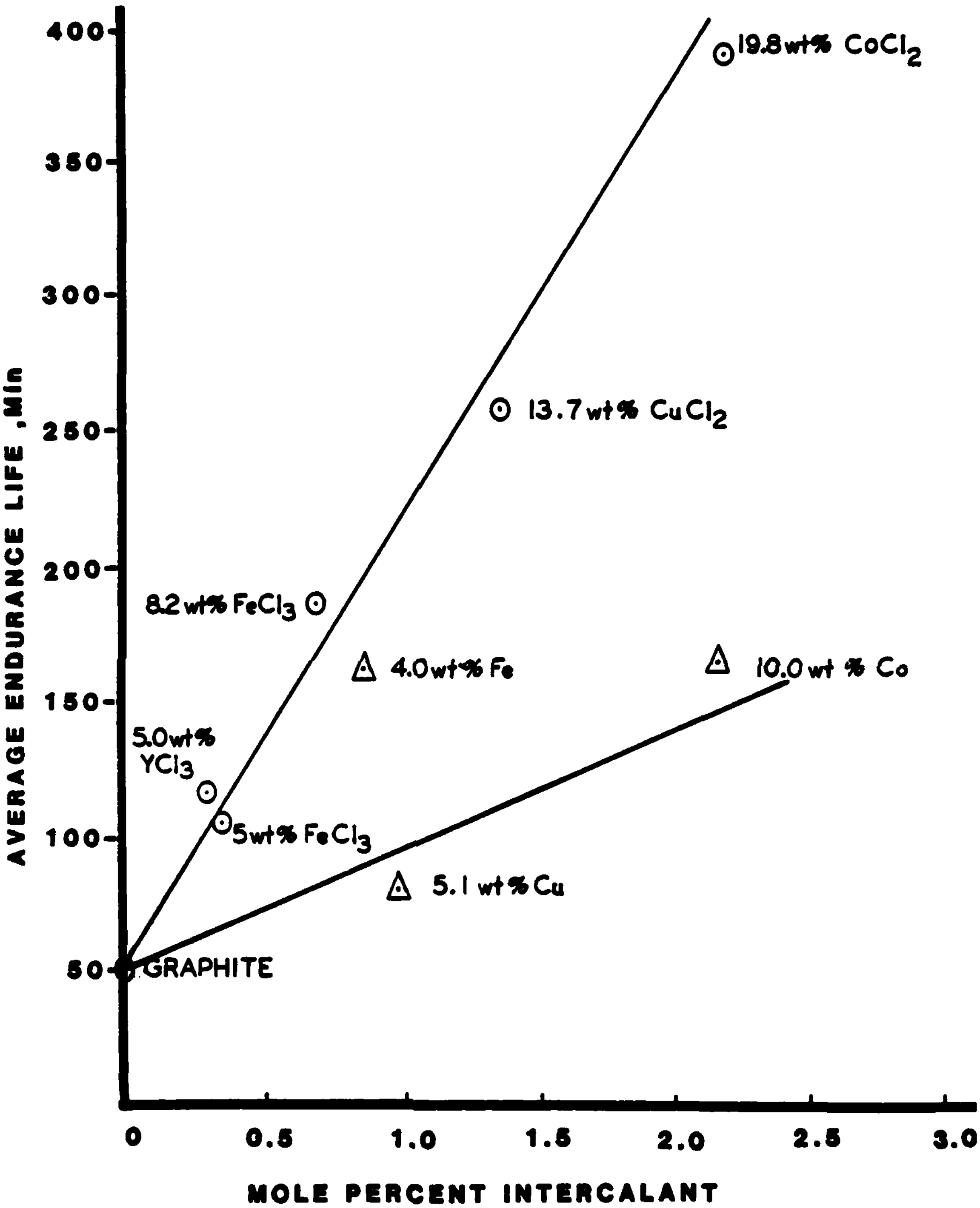

FIGURE 2 EFFECT OF CONCENTRATION OF METAL CHLORIDE AND METAL INTERCALANTS ON ENDURANCE LIFE

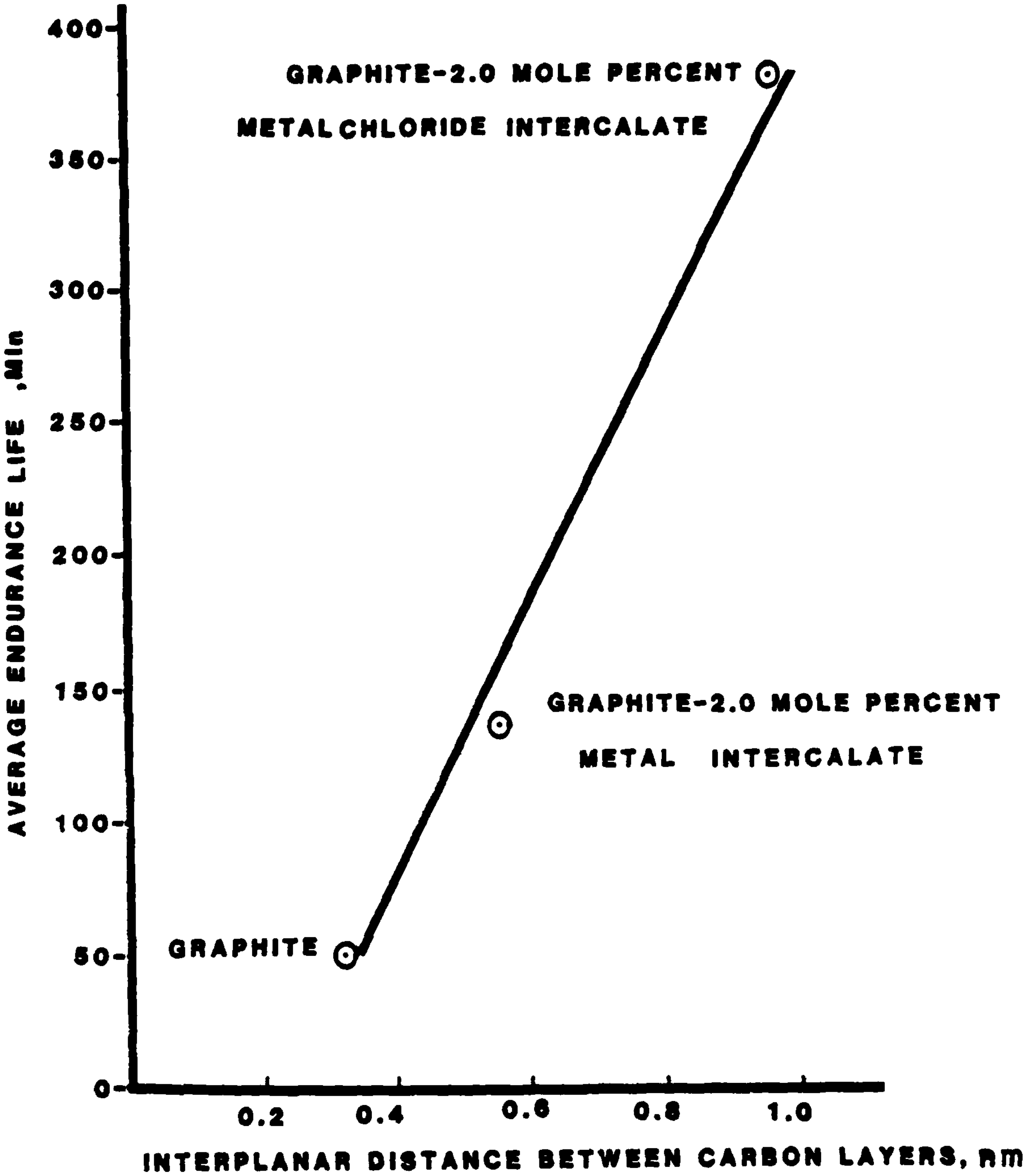

FIGURE 3 EFFECT OF INTERLAYER CARBON SPACING ON ENDURANCE LIFE

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