

## **THERMOGRAVIMETRIC CHANGES INFLUENCED BY THE COMPACTION OF GRAPHITE-CrO<sub>3</sub> INTERCALATION COMPOUNDS**

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The changes resulting from the compression of graphite-CrO<sub>3</sub> intercalation compounds are demonstrated in the TG curves. In comparison with the samples examined in the form of a flake bed, the compacted compounds begin to decompose at lower temperatures and their weight loss is higher, particularly above 220 °C. To explain the obtained results, the pressure-induced changes in the structures and the activities of the compounds are considered in relation to the method of intercalation, the concentration of the intercalant and the extent of exfoliation.

Intercalation compounds of graphite (GIC) are formed in reactions between graphite and certain elements or molecules. The alternating sequence of *n* hexagonal graphite layers and a monolayer of foreign species, called the stage number, is a characteristic feature of their structure. The insertion of the guest species between the host graphite interspaces results in the creation of new, interesting properties. The structural, electrical and thermal changes effected by intercalation have been the most intensively explored [1, 2].

The thermal studies have often related to the exfoliation phenomenon caused by the thermal decomposition of GIC [3–9]. Above a critical temperature, the intercalant escapes from the graphite interspaces, as demonstrated by a large expansion of the sample along the *c*-direction and consequently a very swollen product is formed. The process of deintercalation can be observed in the TG curves if the released intercalant itself undergoes thermal decomposition and/or participates in a secondary reaction with the graphite carbons. The occurrence of both processes has been reported for graphite-CrO<sub>3</sub> intercalation compounds (GIC-CrO<sub>3</sub>'s) [10–12]. When flakes of GIC-CrO<sub>3</sub>'s are heated above 220°, lower oxides of chromium are formed, not only due to the thermal decomposition of the intercalated CrO<sub>3</sub>, but also as a result of the reduction of CrO<sub>3</sub> by the graphite carbons. Consequently, a two-phase mixture of these oxides and the depleted

graphite is formed. However, it has recently been demonstrated for GIC-CrO<sub>3</sub>'s produced in the dry and impregnation-dry methods that the formation of lower oxides also occurs at temperatures distinctly lower than that mentioned above [13]. In this case, lower oxides assumed to be a partially reduced form of the intercalated CrO<sub>3</sub> are still linked to the graphite lattice. In a somewhat earlier paper [12], it was reported that CrO<sub>3</sub> can also be deintercalated from the same compounds without any change in the oxidation state, if the compound flakes are subjected to compression. In this case, the departure of Cr(VI) from the compound can result either from the weakness or from the complete disappearance of bonding between the intercalant and the carbon atoms. There is also the possibility that intercalant islands trapped within the structural pockets are released under pressure. The latter effect is observed more often for the compounds prepared from graphite flakes than for those from HOPG (high-oriented pyrographite) or graphite crystal. The explanation for this lies in the fact that not all flakes are positioned exactly parallel to the bottom of the die plate, and thus some of them are forced parallel instead of perpendicularly to the graphite layers. It is therefore clear that these layers can be mutually moved or even crushed, releasing intercalant.

Up to now, no attention has been paid to the thermal properties of the compacted flakes of GIC's. This paper provides the first contribution to the thermal weight loss of compacted flakes of GIC-CrO<sub>3</sub>'s. The TG curves are supplemented by the thermal exfoliation data.

## Experimental

### *Materials*

The intercalation compounds were prepared from 99.7 wt% C graphite flakes kindly provided by Graphitwerke Kropfmühl AG, West Germany. The compound denoted Al-WI (Sample 1) was prepared from material provided by the producer, including the particles with diameters ranging from 67 to 1440  $\mu\text{m}$ , whereas the other compounds were obtained by using the sieve fraction of 170 to 283  $\mu\text{m}$ . The sieve analysis data on the graphite used have been reported previously [12].

The intercalation compounds were prepared by the dry, the impregnation-dry and the solvent methods. The first two methods have already been described in detail [12, 14]. The intercalation by the solvent method was carried out according to the procedure of Platzer [15], using a mixture of 1 g graphite and 5 g CrO<sub>3</sub> in 50 cm<sup>3</sup> glacial acetic acid. The preparation, the chemical results and the X-ray data are listed in Table 1.

The cylindrical compacts of the compounds with a diameter of 13 mm were

**Table 1** Chemical analysis, X-ray data and weight loss of flakes of the graphite-CrO<sub>3</sub> intercalation compounds

Sample	Method of intercalation	(IC = Cr/C) × 10 <sup>3</sup>	Main graphite and intercalation lines		Weight loss			
			<i>d</i> , Å	<i>I</i> / <i>I</i> <sub>0</sub>	at 170 °C		at 280 °C	
1	2	3	4	5	6	7	8	9
A1-W1	Impregnation-dry [12, 14]	35.6	3.35	100	0.10	2.81	2.12	59.5
A2-S1	Dry [10, 17]	10.4	3.52*	17	0.04	3.85	0.80	76.9
A2-AC	Solvent [15]	22.8	3.35	100	0.22	9.65	0.67	29.4
			3.60	95				

\* This broad peak appearing in the range 3.51–3.62 Å is probably composed of many components of mixed stage structures.

\*\* This peak was about three times higher before intercalation

produced by using a stainless steel die. To align the compound flakes parallel to the bottom piston, a die filled with intercalation compound was shaken for some time by a vibrator at a frequency of 50 Hz. The die was filled with a mass of the compound giving a compact about 4 mm thick. The precise mass of samples of 800 mg was achieved by finishing with abrasive fibre. The compacts were formed under a pressure of 400 MPa. The compacts were under pressure for 0.5 h and were then removed from the die.

### Methods

X-ray diffractometer recordings were obtained by using filtered  $\text{CuK}_\alpha$  radiation.

The thermogravimetric measurements were made in argon ( $50 \text{ cm}^3 \text{ min}^{-1}$ ) with a MOM OD-102 derivatograph. The platinum plate served as the support for the compacted samples, whereas a platinum crucible was used for the flake bed samples of 800 mg. On account of the crumbling of the compacts occurring near  $300^\circ$ , the measurements were carried out in the range  $20\text{--}280^\circ$ . All measurements were performed at a heating rate of  $10 \text{ deg min}^{-1}$ . To determine the extent of exfoliation, the thickness of the compacts was measured at 20, 170 and  $280^\circ$  during separate runs.

### Results and discussion

Intercalated  $\text{CrO}_3$  behaves in a different way from  $\text{CrO}_3$  reactant. It has been shown [10, 12, 14, 16] that the DTA curves of the intercalation compounds do not demonstrate the endothermic peak associated with the melting of  $\text{CrO}_3$ . This fact was attributed to the changes in energy bonding of  $\text{CrO}_3$  as a consequence of intercalation. However, above  $220^\circ$  a characteristic exotherm with peak at about  $320^\circ$  was observed for  $\text{CrO}_3$  alone, as well as for GIC- $\text{CrO}_3$ 's. The decompositions of the compounds prepared by the dry and the impregnation-dry methods were studied earlier in the temperature range  $220\text{--}320^\circ$  [12]. It was shown that the weight loss of the compounds results from the decomposition of  $\text{CrO}_3$  alone, and from its interaction with graphite carbons. On the other hand, there was evidence that this parameter is not simply proportional to the amount of intercalated  $\text{CrO}_3$ , but also depends on the method of intercalation and the kind of graphite. The complete TG curves presented in the present work (Fig. 1) demonstrate that the decompositions of GIC- $\text{CrO}_3$ 's begin at temperatures distinctly lower than  $200^\circ$ . The compound A2-AC made by the solvent method is thermally unstable even below  $100^\circ$ . This result is in agreement with that of Metz and Meyer-Spasche [11], who found  $\text{CO}_2$  to be the product of the decomposition. Comparison of the weight loss from Fig. 1

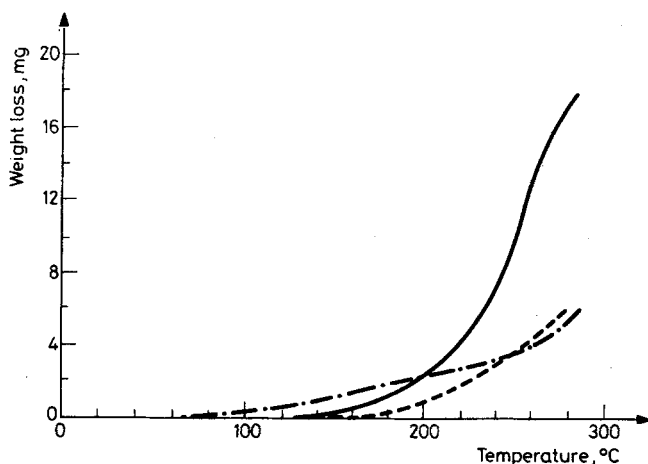
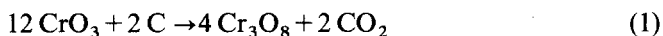


Fig. 1 TG curves of the flake bed of the graphite- $\text{CrO}_3$  intercalation compounds recorded in argon. Sample weight: 800 mg. — compound A1-W1 ( $\text{C}/\text{Cr} = 28.1$ ); --- compound A2-S1 ( $\text{C}/\text{Cr} = 96.4$ ); -.-.- compound A2-AC ( $\text{C}/\text{Cr} = 43.8$ )

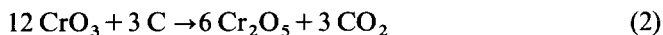
with the intercalant concentration, IC, taken as the ratio  $\text{Cr}/\text{C}$ , did not reveal a simple proportion (Table 1). Figure 1 shows that the beginning of weight loss for the compounds of the dry and the impregnation-dry methods is shifted towards higher temperatures as compared with the compound made by the solvent method. As can be seen from Table 1 (columns 7 and 9), the weight loss,  $\Delta m$ , related to the intercalant concentration, IC, is the highest for compound A2-AC below the intercalation temperature (e.g.  $170^\circ$ ), but for compound A2-S1, containing the lowest amount of intercalant, above the intercalation temperature (e.g.  $280^\circ$ ). To understand this curious behaviour, X-ray data should be taken into consideration. Table 1 (columns 4 and 5) shows that the most perfectly ordered product appeared to be the compound made by the solvent method. A broad and flat peak attributed to intercalation in the range  $3.51\text{--}3.62 \text{ \AA}$  was recorded for the compound produced in the impregnation-dry method. Its intensity, however, was low in comparison with the graphite peak at  $3.35 \text{ \AA}$ . These features indicated that this sample is a dilute compound of mixed stages [12]. Finally, compound A2-S1 revealed only diminution of the intensities of lines corresponding to graphite, and no new lines characteristic of intercalation were found. Although the degree of dilution of the latter compound is undoubtedly higher than those of the other ones, the exfoliation is still observed for the particle sample as well as the compact. This property shows that compound A2-S1 is also an intercalation compound. The above consideration lead to the conclusion that the temperature of the beginning of weight loss depends more on the structural properties of the compound following from the intercalation

method than on the intercalant concentration. The disordering of compounds A1-W1 and A2-S1, possibly with intercalant existing as islands trapped within graphite cages or at grain boundaries [18, 19], hampers the reaction between intercalant and graphite carbons. At above 200°, the thermal changes in weight loss may be expected to depend more on the intercalant concentration, because of the thermal decomposition of CrO<sub>3</sub> itself [20, 21]. Indeed, the position of the curves in Fig. 1 is in agreement with this assumption. However, the weight loss  $\Delta m$  can not be strictly proportional to the intercalant concentration IC, because of competitive reactions taking place between the intercalant and graphite carbons [12]. The rates of these reactions can depend on the activity of the intercalated CrO<sub>3</sub> and also the activity of the carbon planes surrounding the intercalant. For example, the reduction reactions can proceed to the formation of lower chromium oxides in different oxidation states, and hence yield a different weight loss:



with  $\Delta m = 7.2\%$ ,

or



with  $\Delta m = 10.7\%$ .

The above mechanisms are supported on the assumption that all molecules of intercalated CrO<sub>3</sub> take part in the reduction reactions. However, there is the possibility that, depending on the microstructural hindrances, only some of the intercalated molecules of CrO<sub>3</sub> with suitably high activity react with graphite carbons to give lower oxides in the same oxidation state. It is not possible to point out at present which of the above mechanisms predominates because, when the compounds are heated over 200°, the exfoliation process occurs in addition. This phenomenon gives rise to a gradual deintercalation as well as an increase in the activity of the graphite layers due to their puckering. The latter effect contributes to the further weight loss. Exfoliation is associated with the rupture of the graphite cages retaining the intercalant islands [5, 22]. The formation of the disordered structures of compounds A1-W1 and A2-S1 can be interpreted via the assumption that the reaction of CrO<sub>3</sub> reactant and graphite occurring during the dry and the impregnation-dry processes of intercalation give rise to the formation of microstructural traps in which intercalant is sealed. On exfoliation, the graphite cages are burst, due to the fact that CrO<sub>3</sub> decomposes to a lower oxide and oxygen, and consequently the released oxide can react more easily with the puckered graphite layers. This would account for why the parameter  $\Delta m/IC$  calculated for 280° is higher in the case of the considered compounds than for the better ordered compound A2-AC made by the solvent method.

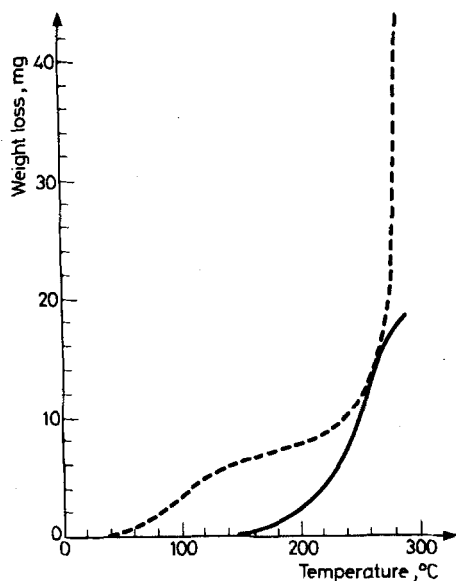


Fig. 2 TG curves of the loose and compacted flakes of the graphite- $\text{CrO}_3$  intercalation compound A1-W1 recorded in argon. Sample weight: 800 mg. — loose flakes; --- compacted flakes

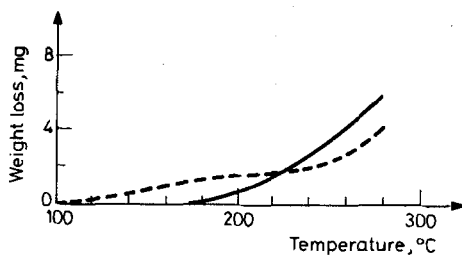


Fig. 3 TG curves of the loose and compacted flakes of the graphite- $\text{CrO}_3$  intercalation compound A2-S1 recorded in argon. Sample weight: 800 mg. — loose flakes; --- compacted flakes

The effects of pressure treatment on the TG curves of GIC- $\text{CrO}_3$ 's are illustrated in Figs 2-4. As a rule, weight loss of the compacted samples is observed at lower temperatures than for the samples in the form of a particle bed. If we assume as above that weight loss at a temperature considerably lower than the intercalation temperature is associated with the reaction between intercalant and graphite carbons, then the question must be raised as to the changes effected in the compounds by the compression, which facilitate the decomposition of the compacted compound. To explain this problem, it is reasonable to return to the results reported earlier on the pressure deintercalation of GIC- $\text{CrO}_3$ 's [12]. Cold

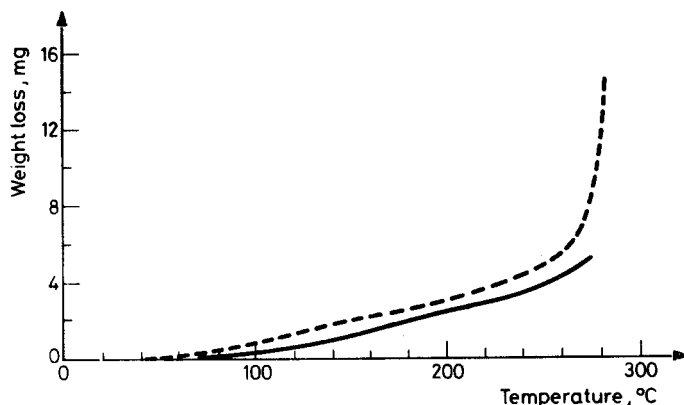


Fig. 4 TG curves of the loose and compacted flakes of the graphite- $\text{CrO}_3$  intercalation compound A2-AC recorded in argon. Sample weight: 800 mg. — loose flakes; --- compacted flakes

water was capable of leaching out part of the intercalant from the compacted compounds in contrast to unpressed ones, which were unaffected even by  $\text{HCl}$  or  $\text{H}_2\text{SO}_4$ . An interpretation which makes this behaviour clear is that, due to the pressure treatment, displacements take place around the dislocation retaining intercalant and hence some of the intercalated graphite cages are partially or entirely opened. In the case of the opened cages, the equilibrium of the reaction between the intercalant and graphite carbons (see Eqs 1 and 2) is shifted to the right due to the decreasing pressure of  $\text{CO}_2$  inside the cages, and therefore the reaction starts at lower temperature. The displacements of the graphite layers forming the dislocation are very probable, because they are not positioned precisely perpendicularly to the action of the force. This arises from the fact that the graphite used is not a single-crystal, but is composed of many crystallites stacked in a non-parallel way between each other. On the other hand, it must be emphasized that, during the compaction, some of the graphite flakes are not positioned perpendicularly to the die piston, and consequently the force acts parallel to the basal planes of the graphite. In the extreme case, the compound flakes are crumbled, which leads to the complete deintercalation of the  $\text{CrO}_3$  as well as a diminution of the flake size. The presented model of the structural changes effected in the compacted samples accounts not only for the lowering of the temperature at which the compound starts to decompose, but also for the increase in weight loss recorded in the TG curves. It was found previously [12] that the weight loss of  $\text{GIC-CrO}_3$ 's was higher when the compounds were prepared from finer graphite. The appearance of the graphite layers with increased activity on the compaction of  $\text{GIC-CrO}_3$ 's results from the exposure of new peripheral areas of graphite, due either to the disruption of the



graphite cages containing intercalant or to the crumbling of the flakes. As regards the pressure effects, it must be noted that, due to the compression, only part of the  $\text{CrO}_3$  is deintercalated and the rest remains in the compound. This fact emerges from the following experiment. When the compact was washed completely free from Cr(VI) ions with hot water, and then milled and subsequently immersed in water, new portions of intercalant were dissolved. On repetition of this treatment, the amount of deintercalated  $\text{CrO}_3$  increased. This experiment showed that, on the first compaction, only a small part of the intercalant is released from the compound. It is reasonable at this point to assume that the remaining intercalant, especially that localized in the more inner regions of the horizontally positioned flakes, can, under pressure, be more firmly sealed in the graphite cages. This fraction of intercalant does not contribute to the increase in the activity of the compound understood as above, but is a potential source of exfoliation. The increase in weight loss is markedly pronounced for compound Al-WI with the highest concentration of intercalant (Fig. 2). As mentioned above, this compound was obtained from the coarse graphite, in contrast to the finer fraction used to prepare the other samples. A comparison of the results for all compounds shows that the parameter  $\Delta m/IC$  likewise has the highest value for compound Al-WI (Table 2, columns 3 and 4). This fact suggests that there are other factors, maybe indirectly dependent on the intercalant concentration, which play a role in the mechanism of decomposition of compacted GIC- $\text{CrO}_3$ 's. A good correlation between the parameters  $\Delta m$  and  $\Delta l$  (the expansion of the compact, see column 5 of Table 2) indicates that the exfoliation phenomena should be taken into consideration. The exfoliation of GIC- $\text{CrO}_3$ 's is effected by the gas products escaping violently from the compound due to the thermal decomposition of the intercalated  $\text{CrO}_3$  or/and its reduction by graphite carbons. Consequently, a very swollen product is formed, as a result of the multiple splitting of the flakes. The reason for the great expansion produced by exfoliation is the formation of very corrugated subflakes which are pinned up between each other. The exfoliation makes the compounds more active [23]. It has been seen, that except for the above-mentioned structural changes caused by the compression, the exfoliation resulting from the release of  $\text{CO}_2$  (Eqs (1) and (2)) can, in addition, increase the weight loss. The exfoliation occurs spontaneously at temperatures above  $220^\circ$ , as this is above the point of decomposition of  $\text{CrO}_3$  itself [20, 21]. The exfoliation effects taking place in GIC- $\text{CrO}_3$ 's above  $220^\circ$  were reported earlier [10, 12]. In light of the above facts, the rise in all curves demonstrated in the present paper in the range  $220$ – $280^\circ$  is quite clear. The intriguing question however is, why the rate of weight loss, especially for compounds Al-WI and A2-AC, is higher after compaction of the flakes. This seems to be a paradoxical feature, because of the additional constraints effected by the compression, which counteract the formation of a puffed-up material with

increased activity. This discrepancy can be suppressed if we take into account the above assumption that some of the graphite cages containing intercalant are more closely tightened on compression. Above a critical temperature, the intercalated  $\text{CrO}_3$  decomposes to  $\text{Cr}_3\text{O}_8$  [20], according to the following equation:



with  $\Delta m = 5.33\%$ .

Oxygen leaves the compound most readily along the grain boundaries and the opened dislocations. However, if oxygen is produced inside the sealed cages, then its release becomes more difficult and its pressure increases until the mechanical strength of the cage walls is exceeded. Prior to the opening of the cages by the forcible action of oxygen, the secondary reaction of the oxidation of graphite can occur [24]:



Since the rate of this reaction increases when the pressure of oxygen increases, the presence of the sealed cages in which the decomposition of the intercalant takes place will accelerate the formation of  $\text{CO}_2$ . It can be seen that the overall reaction resulting from Eqs (3) and (4) is reaction (1), with a higher weight loss than that given in Eq. (3). It is worth noting that, as mentioned above, the reactions taking place above  $220^\circ$  can also involve the reduction of  $\text{CrO}_3$  according to Eq. (1). Moreover, there are other reasons which can contribute to an increase in the weight loss of the compacted compounds. If the local overheating caused by the exothermic reaction (4) raises the temperature to about  $300^\circ$ , an additional weight loss will take place according to the following reaction [20]:



It has been pointed out by Hennig [25] and Volkow [26] that the rate of oxidation of the graphite edges is distinctly higher (between 4 and 100 times) than the oxidation rate perpendicular to the graphite layer surface. From this, it can be deduced that, during the oxidation reaction Eq. (4), the graphite cages are extended mainly along the graphite layers, and consequently their lateral opening can take place. Table 2 shows that the weight loss of the compacts runs parallel with the exfoliation represented by the parameter  $\Delta l$ . The expansion is particularly large at  $280^\circ$ , when the rate of the decomposition of  $\text{CrO}_3$  has the highest value. The curves obtained for compounds A1-W1 and A2-AC (Figs 2 and 4) exhibit a pronounced weight loss of the compacted samples above  $220^\circ$  in comparison with the loose flake samples, which is an accordance with the mechanism considered above. From this respect, the compound made by the dry method (A2-S1) behaves non-typically (Fig. 3). It must be taken into account however, that this compound is the most dilute and it is

**Table 2** Weight loss and exfoliation extent of the compacted graphite-CrO<sub>3</sub> intercalation compounds

Sample	(IC = Cr/C) × 10 <sup>3</sup>	At 170 °C			At 280 °C		
		$\Delta m$ , %	$\Delta m/IC$ , %	$\Delta I^*$ , %	$\Delta m$ , %	$\Delta m/IC$ , %	$\Delta I^*$ , %
1	2	3	4	5	6	7	8
A1-W1	35.6	0.80	28.09	19.7	5.40	151.68	114.0
A2-S1	10.4	0.14	13.46	1.9	0.55	52.88	11.8
A2-AC	22.8	0.29	12.72	7.2	1.80	78.94	36.1

$$* \Delta I = (I_t - I_{20})/I_{20}$$

likely that the preceding increase in weight loss of the compact in the range 100–200° consumed an excessively large amount of the intercalant to be decomposed above 220°.

In conclusion, the present work has shown the distinct changes caused in the TG curves by compression of the GIC-CrO<sub>3</sub>'s. The pressure influence is pronounced in the case of the compounds prepared by the impregnation-dry and the solvent methods, in spite of the different features characterizing these compounds, such as the size of the graphite flakes, the intercalant concentration and X-ray data. This is evidence that there are other common properties responsible for the thermal behaviour of the GIC-CrO<sub>3</sub>'s, which should be clarified in the future.

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**Zusammenfassung** — Anhand von TG-Kurven wurden Veränderungen demonstriert, die durch das Verdichten von Graphit-CrO<sub>3</sub>-Einlagerungsverbindungen hervorgerufen werden. Im Vergleich zu Proben, untersucht in Form von Flocken, beginnen die kompakten Verbindungen sich bei niedrigeren Temperaturen zu zersetzen und zeigen einen grösseren Masseverlust, insbesondere beim Überschreiten von 220 °C. Zur Deutung der erhaltenen Ergebnisse wurden die druckverursachten Veränderungen in Hinsicht auf das Verfahren der Einlagerung, die Einlagerungskonzentration und auf das Ausmass der Abbröckelung in Betracht gezogen.

**Резюме** — На кривых ТГ показаны изменения, вызванные сжатием интеркаляционных соединений графит-CrO<sub>3</sub>. По сравнению с соединениями, исследованными в виде чешуйчатых пластинок, спрессованные соединения начинают разлагаться при более низких температурах и с большей потерей веса, особенно при прохождении температуры 220°. При объяснении вызванных давлением изменения структуры и активности исследованных соединений, было принято во внимание метод интеркалирования, концентрация интеркалирующего агента и степень расслоения.