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PLOTTING OF PHASE DIAGRAMS FROM DTA DATA

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ABSTRACT

In order to establish the optimal conditions for plotting phase diagrams, using DTA data, the system naphtalene-β- naphtol and naphtalene -α- naphtol have been investigated.

INTRODUCTION

Phase changes in solid-liquid system are accompanied by considerable thermal effects easy measurable through DTA.

This is the reason for phase diagrams can be plotted by using DTA technique[1,2].

EXPERIMENTAL

The DTA curves have been recorded by help of a MOM Budapest type Paulik-Paulik-Erdey Q-1500 derivatograph, using α-Al_2O_3 as reference material.

RESULTS AND DISCUSSION

Due to the nonuniform distribution of temperature and to the instrumental factors the DTA signal is not located at the equilibrium temperature, but in a temperature interval. The initial temperature (T_i), the maximum temperature (T_m) and the final temperature (T_f) have been graphically determined according to the ICTA recommendations[3].

In figure 1 the dependences of T_i, T_m and T_f upon the weight of sample for the melting of naphtalene in platinum covered crucible with the volume of 1 cc and a heating rate 1,2°C/min are given. One can notice that the value of T_f and T_m are more sensitive of the change of weight than the T_i value.

For the samples with the weight of 4o mg, T_i is close to 8o°C, temperature which corresponds to the melting of naphtalene in equilibrium conditions and the DTA signal exhibits an

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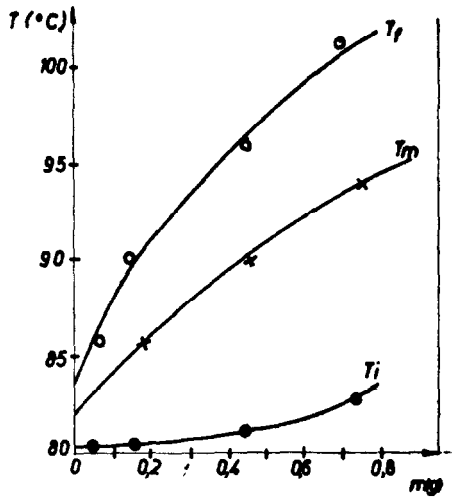

Figure 1

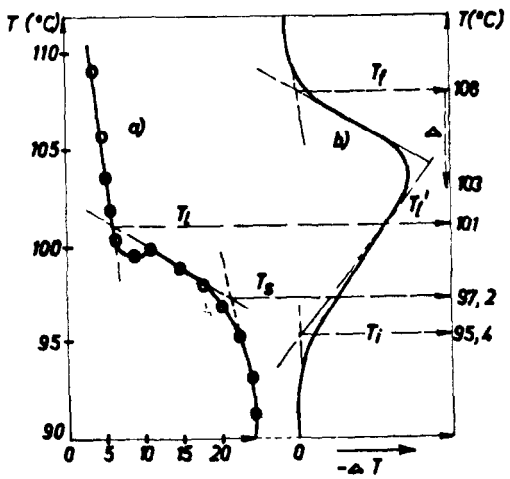

Figure 2

instrumental width $\Delta = T_f - T_i = 5^{\circ}C$. The lowering of the sample weight and heating rate limited at m=4o mg and $\beta = 1,2^{\circ}C/min$ by the sensibility of the apparatus.

A comparison between the classical cooling curve (a) and the DTA curve (b) for the naphtalene-β- naphtol samples with the naphtalene mole fraction X_N=o.56 is given in figure 2. The temperature T_i(DTA) can be assimilated with the temperature T_s(solidus) on the cooling curve. The high difference between T_f(DTA) and T_l(liquidus) is due to the temperature gradient from the DTA system[4] as well to the recording of the DTA curve (b) during heating and of the curve (a) during cooling.

From figure 2 it turns out that $T_l \simeq T_f - \Delta$.

The curves of T_i, T_l (dotted) and T_f (DTA) versus composition (figure 3) show that naphtalene and β -naphtol are soluble no matter of their contents.

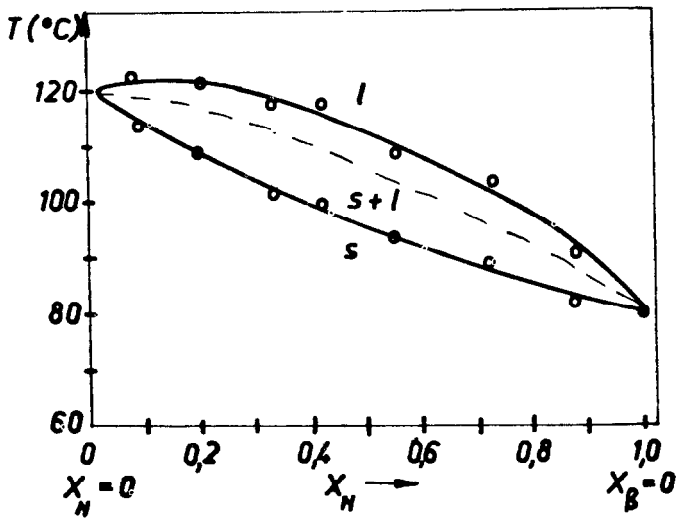

Figure 5

The system naphtalene - - naphtol exhibits two melting DTA peaks. The location of the first peak ($T_i=60^oC$) does not depend on composition, thus the first peak belongs to an eutectic. The other peak is due to the melting of the solid solution in excess. From the Tammann's diagram one finds for the eutectic $X_N=0.6$; $X_\alpha=0.4$. For the heat of melting of the eutectic one finds $H_E=38$ cal/g.

CONCLUSIONS

1. The DTA method for plotting phase diagrams exhibits the following advantages with respect to the method of the cooling curves:

a) it allows the avoidance of the undercooling when used during the temperature increase

b) it allows the determination of the eutectic latent heat of melting.

c) it can be used for the investigation of the nonequilibrium solid phases

2. Among the desadvantages of the method one has to notice:

a) the DTA phase diagrams should be corrected for the instrumental width of the signal

b) the solid samples should be treated in a standard way due to the sensitivity of the results to their history

c) the temperature gradients can not be avoided.

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