

THERMAL ANALYSIS APPLIED TO ELECTRONIC MATERIALS AND PROCESSES

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ABSTRACT

The brief survey is intended to describe some of the recent applications of thermal analysis in the field of electronics and communications, particularly those described at the 8th ICTA. Virtually every type of material is used in some manner within this broad topic, however, we will limit ourselves to metals, semiconductors, ceramics, and polymers having direct application to electronic and optical devices. Attention will also be given to the selection of examples which indicate the wide range of techniques encompassed by the general term, thermal analysis.

METALS

One of the major uses of thermoanalytical methods has traditionally been in the field of phase equilibria. Information regarding both the equilibrium and non-equilibrium phase diagrams is of fundamental importance to all areas of materials science. From the viewpoint of electronic applications no phase diagram of a metallic system could be more important than Pb-Sn, the basic solder system. The recent work of Kuck [1] not only illustrates the use of DSC techniques to determine the liquidus and solidus curves within the important compositional range of that system, but also utilizes the techniques to solve real problems concerning the heterogeneity of solder deposits and further demonstrates how DSC can be used for quality control in a very important technological application.

The precipitation processes control the desirable mechanical properties of Cu-Be alloys which make them useful in relays and switches. Again DSC methods prove valuable as shown by Ohshima and Hirano [2]. They investigated the kinetics of the complex multistep precipitation hardening phenomena and verified their model by further TEM and electron diffraction studies.

Thermomagnetometry is a valuable technique to study the magnetic properties of various alloys [3]. For example, it has been used to study the

nature of the spinodal decomposition that imparts the highly desirable magnetic properties to chromindur alloys [4]. Similarly, Illeková, et al. [5], have used TM to monitor the structural relaxation in Fe-B metallic glasses. A more general survey of thermomagnetometry is also included in this issue [6].

The general area of metallic glasses has been a fruitful field for thermo-analytical studies and many investigations have been made using DSC [7] and electrical conductivity [8] techniques to study the glass transition, crystallization, annealing, and other phenomena so important in these materials. Lukač et al. [9] have utilized a rather unique method of first implanting radioactive ^{85}Kr into the sample and then following the radioactivity of the sweep gas during subsequent heating similar to emennation thermal analysis. In this manner the temperatures associated with the various relaxation and crystallization processes were determined.

Other areas of the application of thermal analysis involve the numerous studies of oxidation and corrosion of metals, generally by following the weight change in the appropriate atmosphere, and studies of impurities such as the evolved gas analysis of gold-plated films employed as electrical contacts [10].

SEMICONDUCTORS AND SEMICONDUCTOR PROCESSING

The technology associated with silicon integrated circuits is largely concerned with the buildup of devices on a silicon wafer by means of various thin film depositions to form several active, passive, and processing layers. The application of thermoanalytical techniques to such processes is frequently limited by the small amounts of the interesting substance relative to the massive substrate. It is necessary to either extrapolate the thin film properties based upon behavior of the bulk materials or to utilize very sensitive techniques.

Mass spectroscopic evolved gas analysis (EGA) is a good example of such a sensitive method. It has been applied to study the problems associated with blister or bubble formation in conducting layers of TaSi_2 [11]. Trapped or occluded Ar was identified by EGA as the source of these blisters and the EGA was used to monitor the Ar content in studies with varying process parameters to minimize the quality of Ar incorporated in these sputtered films.

Figure 1 [11] shows an EGA plot of the evolution of Ar (AMU 40^+) from a sputtered film of TaSi_2 . The evolution at high temperature during subsequent processing is what leads to blister formation. The method has also found use in demonstrating that the quantity of oxygen evolved at relatively low temperatures from films of Ta_2O_5 prepared by reactive sputtering is

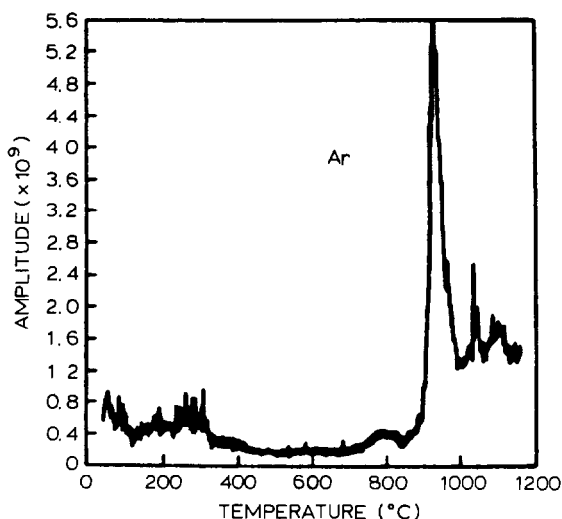


Fig. 1. EGA curve for Ar in sputtered TaSi₂ [11].

directly related to the dielectric properties of these films [12]. The more oxygen evolved around 300°C the greater the dielectric loss of the film.

Two examples in which experiments were performed on the bulk material and assumed to be directly relevant to thin film applications are (1) the properties of BPG, Boro-Phosphosilicate Glass, used as an insulating layer and for the final passivating coating in VLSI structures [13] and (2) the thermal decomposition of Au [14] or Pd [15] compounds by a laser beam in order to scribe conducting or opaque paths of the metal.

The newer generation of semiconductor devices may be based upon III-V materials rather than Si because of the greater device speed attainable. In addition there is great interest in semiconducting lasers and LED light sources based on these materials. Because many of these devices are prepared from the melts, such as by liquid phase epitaxy (LPE), there is great interest in both the phase equilibria and the thermal stability in such systems. Bagirov et al. [16] have determined by DTA and thermodynamic calculations the liquidus curve within the very important (Ga, In)/(As, Sb) system. InP single crystal is frequently used as substrate material for subsequent LPE growth of the active films. The thermal stability of this material is important during the processing. Highly sensitive mass spectrographic EGA, see Fig. 2, was used successfully to study the early stages of the thermal degradation of InP [17]. The thermal properties and phase equilibria of many of these important III-V materials have recently been assembled and critically appraised in several review articles [18,19].

Making stable electrical contacts to these III-V semiconducting materials is frequently a source of problems. The ohmic nature of the contacts is

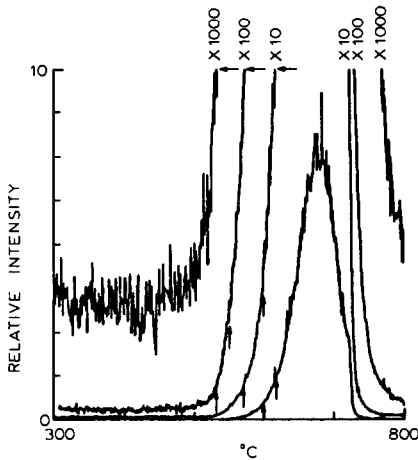


Fig. 2. EGA curve for P_2 from InP at various amplifications [17].

sometimes destroyed at temperatures of subsequent processing or use. Again mass spectroscopic EGA techniques have been very helpful in understanding the nature of the problem and indicate the temperatures at which these mechanisms of degradation become important [20–22].

The large number of papers [23–36] presented at the 8th ICTA on the topic of vitreous chalcogenide semiconductors is indicative of interest in these materials for a wide range of uses, e.g., semiconductor devices, photovoltaic materials, inorganic photo-resists, piezoelectric properties, long wavelength optical transmission media, etc. Most of the major thermoanalytical techniques along with X-ray diffraction have been used in these studies.

CERAMICS, GLASSES, AND ENERGY RELATED MATERIALS

In order to meet the greater demands being imposed on communications systems, the trend is toward optical systems with glass fibers replacing the traditional cables of electrical conductors. While tremendous success has been achieved with fused silica fibers, there is still an enormous effort being aimed at achieving low loss transmission at longer wavelengths and developing new and less expensive processes. Heavy metal fluoride glasses are one of the potential material considered as the transmission medium using longer wavelengths. In determining the glass transition temperature and crystallization temperatures and kinetics, DSC techniques have been invaluable in establishing the working range and conditions for forming and drawing such glasses [37].

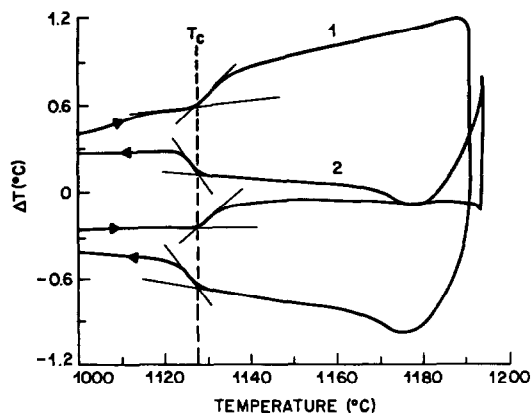


Fig. 3. DTA curves for two samples of crushed congruent crystal of lithium niobate [43].

Sol-gel methods offer interesting, less expensive possibilities for forming glasses as an alternative to the modified chemical vapor deposition process, at least for the less demanding applications. The chemistry of gel formation, nature of the drying process sintering, and the general characterization of these materials present numerous intriguing possibilities for calorimetric and thermoanalytical investigations [38–42].

Besides the semiconducting light source and fiber optic transmission media, it is necessary to modulate, demodulate, and switch these light beams in order to have a communication system. Consequently, there is considerable interest in electrooptical devices and materials. One of the leading candidates among the materials is single crystal LiNbO_3 . The demands upon crystalline perfection and chemical homogeneity are great so that the crystal growth must be done at precisely the congruent composition in order to satisfy these demands. Since LiNbO_3 melts incongruently it is essential to establish the precise non-stoichiometric composition (Li-deficient) at the congruent melting point. The ferroelectric transition temperature (T_c) is fairly dependent upon the Li content of the material and hence is a convenient analytical tool. This transition, however, is a second-order effect which gives rise to a relatively weak indication in the DTA curve, see Fig. 3. Nevertheless, careful DTA and dilatometric studies [43,44] have been used to precisely define the congruent melting composition and T_c as a function of composition. In addition TG has been used to study the subsequent in-diffusion of Li_2O to achieve the stoichiometric or near-stoichiometric compositions [44,45].

There are many other potential applications of thermal analysis to optical communications. Some of these are described in the section on polymers.

An example of other devices which depend upon the unique electrical properties of certain materials are superconducting, Josephson junction

devices. Films of superconducting NbN are very slightly oxidized to provide the insulating barrier of niobium oxide through which the electron must tunnel. This oxidation process is critical and has been successfully studied by TG and X-ray diffraction techniques [46,47].

Applications of thermal analysis to the general topic of the reactivity and characterization of solids are very extensive and many of these affect the synthesis of electronic ceramics. In the "Proceedings of the 8th ICTA" there are numerous examples, primarily related to ferrites. There are basic studies on the particle reactivity of various iron oxides [48,49] and how they react to form ferrites or garnets [50,51]. There are studies on the reactivity of precipitated precursor materials to form various spinels [52,53]. Thermomagnetometry has also been used to follow ferrite formation [6]. X-ray diffraction and electrical measurements are used to follow the site selection of ions within the spinel structure [54]. Once the ferrite has been formed it is essential that the proper valence be established during the sintering process and the subsequent cooling in order for the final device structure to have the desired properties. Isothermal gravimetry has traditionally been used to help determine and maintain the proper stoichiometry [55,56]. The fine plenary presentations by Prof. Jesenák [57] and to some extent by Prof. Boldyrev [58] address this general topic of solid-state reactivity.

The final topic to be briefly considered in the inorganic portion of this survey concerns the subject of energy related materials. The plenary lecture by Dr. Ozawa [59] is a very excellent introduction to the application of thermoanalytical methods to studies of latent heat energy storage. Another interesting report on potential energy storage materials is that by Struble and Brown [60] describing the use of various hydrates of the type $\text{Ca}_3\text{M}(\text{OH})_6\text{X}_3 \cdot 26\text{H}_2\text{O}$ where $\text{M} = \text{Al}, \text{Fe}$ or Cr and $\text{X} = \text{SO}_4$ or CO_3 .

Batteries and fuel cells are another area where thermal analysis has proved useful. Studies of electrode material [61], ionic conductors [62,63], and battery performance [64] are examples.

POLYMERS

Although microelectronics has had an important effect on our lives during the past two decades, many traditional polymer chemists and engineers are not very familiar with the many uses of polymers in the current state-of-the-art of this rapidly developing technology. Here, a brief overview of various polymer applications in the IC industry will be given [65]. Recent examples where thermal analysis of these speciality polymers have been reported at ICTA, NATAS, and other meetings of thermal analysts will be cited.

The key building-block in this multibillion dollar business is the integrated circuit, IC. These devices are simply interconnected layers of transistors, diodes, and other components made directly on a silicon sub-

strate. Elements within the silicon chip have exact three-dimensional geometrical layouts, tailored to the needs of the device. These 3-D patterns are generated on the silicon by photo-engraving (lithography) [66,67]. Photolithography is synonymous with advances in miniaturization of ICs and these patterns are developed from polymers which have been selectively exposed to some type of radiation. The latter step makes the exposed areas either more soluble or less soluble than the unexposed regions. In this manner a well defined pattern can be created as the soluble polymer is etched away.

Individual elements are connected by metallic conductors. An insulating material is placed between metallic lines. Traditionally this material has been an inorganic oxide or nitride. Recent research is aimed at replacing these inorganics with polymer dielectrics, most notably polyimides. Good electronic properties as well as ease of processing make these polymers suitable as insulating layers in multi-level within-chip interconnects.

Lastly the assembled IC must be protected by enclosing it in a suitable material. The lowest-cost packaging materials are plastics. Currently, these encapsulating compounds are thermoset-composites. Typically a silicone or epoxy resin is loaded with silica in order to reduce its coefficient of thermal expansion, CTE, and improve its thermal conductivity. The attempted matching of CTEs between the encapsulant and the silicon device is crucial in trying to minimize stresses which develop during thermal cycling. And as ICs become more complex and denser the encapsulant's ability to transport heat away becomes more important. At this point we have focused on three major uses of polymers in ICs: photoresists, intermetallic dielectrics, and packaging. Polymers also have important roles to play in the development of printed circuit boards, cables, data storage media, and UV-curable optical fiber coatings. All these diverse uses of polymers call for many kinds of characterizations of their material properties, such as curing and expansion behavior, glass temperatures, volatiles, thermal conductivities and long-term aging behavior. Thus, thermal analysis is well suited to playing a crucial role in understanding the critical properties and processes of polymeric materials as they are formulated for today's electronics industry.

Differential scanning calorimetry (DSC) is used to study the reactions of thermosets such as epoxy-novolacs [68,69], amine-cured epoxies [70–72], polyimides [73], epoxy-phenolic magnetic recording inks [74] as well as the photolysis of light-sensitive materials like multifunctional acrylates [75]. The measured heats of polymerization, ΔH_r , are thermodynamic properties whose values change only slightly with temperature. In the absence of other enthalpic events, the DSC ordinate is a direct and linear measure of rate of cure.

Time-temperature-transformation (*TTT*) diagrams for thermosetting resins indicate the difficulty in driving the reaction further after vitrification has occurred and suggest that the glassy material can be reacted further by simply increasing the cure temperature, T_c , above the glass transition

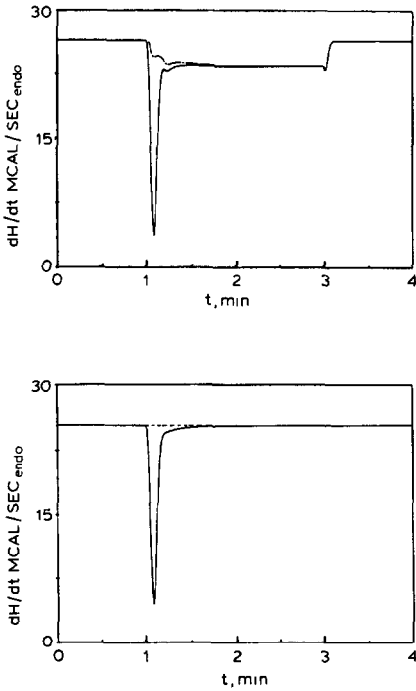


Fig. 4. DSC trace for photopolymerization of a light-sensitive monomer at 25°C. UV lamps on at 1.0 min off at 3.0 min.

temperature, T_g [76]. No current model which utilizes dynamic DSC measurements can predict the curing behavior of a resin when the process is diffusion controlled. For this and other complexities which may arise in thermal or photo-curing reactions, isothermal experiments are advised in the kinetic analysis of DSC data [77].

Appelt and Abadie combined a radiation exposure unit to a DSC to monitor the in-situ photochemical reactions of an acrylate resist [75]. A typical thermogram for an exposed sample is shown in Fig. 4. The rate of reaction is computed from the peak maximum:

$$R_p = (dH/dt)\Delta H_r F$$

with dH/dt , the DSC heat of reaction at the peak maximum; and F , the fraction of unreacted material at the peak maximum. The resulting rate constants are plotted in an Arrhenius plot which yields two straight lines intersecting at 35°C. This particular photoresist exhibits a low activation energy, E_a (1.4 kcal mol⁻¹), above 35°C, which indicates the monomer reaction is following the expected propagation and termination steps. Below 35°C E_a equals 6.2 kcal mol⁻¹ and suggests the process may have become diffusion controlled.

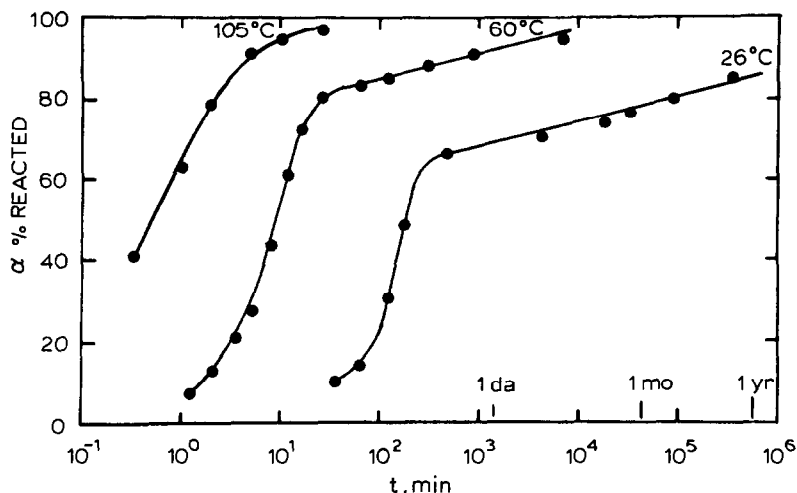


Fig. 5. Apparent degree of conversion versus log time for epoxy curing at 26, 60 and 105°C.

Immobilization of the reacting species in a similar system was reported by Bair where T_g can reach or exceed the cure temperature, T_c , and cause the reaction to slow down dramatically. This leads to entrapped radicals which when the temperature is raised above T_g in a dynamic scan allow the multifunctional monomers to undergo immediate exothermic reaction [78].

Two distinct curing stages for an epoxy thermoset can be seen in Fig. 5. In this case DSC measurements of the amine curing of a DGEBA epoxy resin at three different temperatures (26, 60 and 105°C) are shown [79]. In the initial stage, the reaction continues at a rapid rate ($E_a = 13 \text{ kcal mol}^{-1}$) until T_g rises to equal T_c . During the second stage when T_g exceeds T_c the diffusion of reactants is restricted and the reaction rate ($E_a = 51 \text{ kcal mol}^{-1}$) decreases by more than three orders of magnitude. These studies show that the epoxy resin reacts at a perceptible rate when stored as much as 50°C below T_g .

Thermal degradation of polymers using TG were used to monitor compositional changes in bornyl acrylate monomers during imaging process [80]. The two-step TG profile of a mixture of these acrylate monomers and an epoxy resin was related to the stereochemistry of the monomers.

Thermal mechanical analysis can be used in the expansion mode to measure not only CTE but also to follow the delamination of PCB laminates [81]. The development of stress in a package device during temperature cycling was stimulated by embedding a copper tube in an epoxy cylinder [82]. Stresses were measured with strain gauges. The report suggests stress at the Cu-epoxy interface can be lowered by reducing the epoxy's T_g .

A well known application of DSC and TG is the study of polyethylene oxidation [83,84]. The screening of antioxidants for their effect on polymer

stability is easily accomplished. However, since the testing temperature is normally high ($> T_m$), extrapolation to use temperature is not feasible in many cases. In some cases, low-temperature, long-term tests are monitored by TA methods to determine when other mechanical or physical tests should be made. Recently the oxidative stabilities of several UV-cured acrylate coatings for optical fibers were examined by a number of these thermoanalytical techniques [85].

CONCLUSIONS

It should be immediately obvious to the reader that thermal analysis plays a very important role in virtually all aspects of the synthesis and characterization of the materials and processes which are vital to the electronics and communications industry. Its presence is pervasive and its future assured. What remains as the crucial step is for scientists and engineers to recognize the value of these techniques in the solution of their problems.

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