Dilatometric Methods: Insights of other researchers

E Pavithra*

Associate Professor, Department of Mechanical Engineering, Vel Tech Rangarajan Dr Sagunthala R & D Institute of Science and Technology, Avadi, Chennai, India

*E-mail: epavithrasenthil@gmail.com

Abstract. Measurement of thermal expansion and contraction of a solid material during heating and cooling is done using dilatometer. To obtain best results dilatometers are operated under vacuum or inert atmosphere. The physical and chemical characteristics are inferred from the dilation of the material. It helps to study the properties of the sintered material and the phase transition of the solid material. Few authors also developed a customized dilatometer for their research applications. This paper deals with those different designs, the various material characteristics providing adequate data to make researchers identify the new area of research for the dissimilar materials. The thermo physical chemical property studies based on the dilatometer are reviewed in this paper.

Keyword: Dilatometer, solid material, thermo physical chemical property

1. Introduction

A well known method for thermal analysis of any solid material is by dilatometry. The change in length and the volume is measured when there is a change in temperature. Also the dilatometer provides the observation on the phase transformations using the microstructure Characterization. As per the ASTM E228-06, dilatometer is measured. The coefficients of thermal expansion are determined between 25ºC and 1000ºC. It is estimated between ±5% below 500ºC and ±3% above 500ºC at a heating rate of 3ºC/minute [1]. The single phase quasi crystalline is formed while heating in the isotherm annealing, prolonged heating results synthesized powder quasicrystals. The same is observed in the investigation of dilatometer and DTA [2]. Mostly Dilatometer is categorized into Linear, volume, capacitive, deformation and non-contact types. Among the above all, capacitive methods are sensitive to small changes in length during measurement of thermal expansion and magnetostriction measurements within solids. For various experiments different types of dilatometers were developed namely small dilatometers for magnetic systems, absolute-expansion dilatometers and open-architecture dilatometers for sample mounting arrangement [3]. When compared with X-ray method which estimates correct dimensional changes induced due to thermal variation. Better services are provided when transducing techniques are equipped with sophisticated tools namely high speed photographic techniques, optical interferometer and capacitance change measurement for calibration purpose [4]. Modern dilatometric techniques find wide variety of applications ranging from metals, alloys and engineering; powder metallurgy to polymers; ceramics and glass industries. Soil properties and stratigraphy were evaluated using dilatometer test (DMT) which is a fast and economic tool [5].
2. Insights of Dilatometer

Density measurement was precisely measured by using both dilatometer and traveling microscope with a least count of 0.001 cm. For temperature control, dilatometer is covered with water and kept inside a cylindrical glass [6]. The dilatometer provides the data for the study of the volume change. But it is necessary to consider the kinetics of \( \gamma \) to \( \alpha \) of the sample. So, calculated length change can be deduced as a ‘virtual’ dilatometer signal. But the unmatched results will not provide the sufficient data. Hence plasticity of the material should be studied to obtain the pure phases [7].

To measure the volumetric growth and determine ASR feature, unlike the presence of fly ash motor and concrete samples containing New Mexico Rhyolite (NMR) and Platte River Gravel (PRG) were examined using dilatometer at different alkalinites [8].

![Figure 1. Ghanem (2012)](image)

For processing, dilatometer is kept at a distance of 14 cm from a spectrum lamp of PRK-2 which works under stabilized working conditions. Thermostat baths were used to carry out the process with temperature control of \( \pm 0.05^\circ \) [9]. With solid state reaction, compositions in series \( \{\text{Pb}(\text{Fe}_{0.5}\text{Nb}_{0.5})_3\}^{1-x} - \{\text{PbTiO}_3\}_x \) (\( x = 0 \to 1 \)) were developed. Differential thermal analysis has been studied as a function of \( x \) where the phase shift changes from tetragonal/rhombohedra to cubic structure, where the x-ray dilatometry and bending high temperature ranges from 298-773 K. Thermal expansion and dielectric co-efficient of 0.2 indicates relax or type behavior of lead iron niobate upto 453 K [10]. The effect of isothermal transformation of cold-rolled high strength TRIP steel was investigated using dilatometry. Transformation of temperatures like \( \text{Ac}_1 \), \( \text{Ac}_3 \) and \( \text{Ms} \) were also studied by dilatometry curves [11].
In DIL 805, the longitudinal temperature gradient increases as heating rate increases. To describe the gradient of temperature inside the sample, 2D electromagnetic FEM simulations are deployed to develop a numerical model which consists of transfer of heat from the sample to dilatometer roads as well as heat conduction within the sample [12].

The dilatometer developed by Manoj et al was fabricated by the available sources at the low price and its ease in maintenance. It has got high temperature resolution (1 K, ±1 mm, 10nm). It is verified by NIST sapphire and high purity metals with nickel and aluminum. Model will be helpful for the thermo analytical analysis, all over the world [13].
An advanced dilatometer for variety of adhesives is designed domestically for the study of thermal expansion, polymerization shrinkage, change in volume and cooling contraction. By this new dilatometer, the effects of the cure parameters are widely investigated [14].

Through spray forming Zircon sand reinforced LM13 is fabricated for a length of 25mm. For a temperature range of 29–470 °C the thermal coefficient expansion for the base alloy and its corresponding composite is measured using Netzsch DIL 402 PC dilatometer [15]. Test coupons of Φ 5 × 25 mm were precisely machined and polished for dilatometer test. The specimens were heated up to 1273 K, with rate of heating rate at 1 K/min under argon atmosphere with a purity level of ≥99.999% during the measurement on Netzsch DIL-402C. The change in length as a function of temperature was plotted to understand the phase change [16].

Laser typed dilatometer is used to investigate the effect of distortion of forged steel which is homogenized at 1553K from the ingot for the current study. During phase transformation at the non-recrystallized region consists of coarse austenitic grain with the presence of low carbon and high Nb within the steel matrix. The microstructure observation also reveals the same [17]. In Netzsch dilatometer DIL 402C, a push rod dilatometer, Thermal growth of CVD single crystal silicon was measured up to 1100°C. The resolution was ±0.125nm of the normal growth. It is functioned in the argon atmosphere, at a temperature of 5 K/min with continuous heating and cooling. The repeated experiments provide the average results and thus optimal output is recorded [18].

To determine the temperature of sintering for the pre-alloyed powder 718 under Ar atmosphere, Differential Scanning Calorimetry (DSC) and dilatometric studies were performed. The samples were sintered at 1260–1300°C for a period of 1-3 hrs based on the results obtained from DSC and dilatometric tests [19]. Based on Vickers hardness, metallographic checks and dilatometry trials confirms the Continuous Cooling Transformation (CCT) diagram for the material. To measure the ferrite-to-austenitic change start (Ac1) and finish (Ac3) temperature a Formastor-Digital deformation dilatometer is used, similarly martensite start temperature and bainite start temperatures are also measured using dilatometer [20].

Thermal expansion using dilatometer is used to determine the expansion properties of lead zirconatetitanate–lead nickel niobate ((1−x)PZT–xPNN, x=0.1–0.5) ceramics for a temperature range of −100 to+450 °C [21]. In Bähr DIL 805 deformation dilatometer, direct strip cast, AZ31 magnesium alloy was examined by the flow curves. Between 300°C and 400°C dynamic recrystallization takes place [22].

Slurry which consists of 36.85wt% kaolin/clay, 33wt% water, 17.42wt%, 12.73wt% feldspar along with an industrial commercial deflocculant (Formsil D) was prepared. Using the dilatometer (Netzsch DIL 402 PC), the comparative linear change in length of the slurry was measured up to 1300 °C with 15 min dwell time and at 5 K/min heating and cooling rate [23].

Rockwell hardness, X-ray diffraction, impact toughness and dilatometer was used to investigate the IF (internal friction) of high alloyed martensitic carbon steel. The stage could be distinguished during the measurement. Change of material is dependent on the internal friction peak [24]. Classification of LaAl0.3Ni4.7 films performed using a capacitance dilatometer revealed that the lattice growth was relative to the partial pressure of H2 over the range 0.01–1.3 atm used in this testing [25]. The transformation of the δ → α’t at or above 150 °C is studied for the naturally aged sample or doping with 238Pu by using Netzsch 402C dilatometer with a low-temperature furnace and silica sample tube and pushrod [26]. To find the precise measurements of change in length on co efficient of thermal
expansion of low thermal expansion materials is found using an optical heterodyne interferometric dilatomter for extreme ultraviolet lithography [27].

3. Dilatometry – a Holistic view
Dilatometry thermal analysis is studied on linear and volume matters. Thereby can be used for wide applications. Other researchers have also emphasized on dilatometry. We practically study on varied terms and could be done related to frequency modulation. At the same time, a newer material has to be identified so that we could use dilatometry in all range of sectors and will be used for inventing newer products which will be greatly beneficial. In seismic analysis for construction based work or earthquake work, flat dilatometry can be used to study. Soil pressure, soil properties, mechanical properties like sheer strength and sheer modulus can also be studied using flat dilatometry. Both flat and seismic dilatometry is used for soil based geology study.

4. Summary
Newer materials can be developed based on the thermal properties by using dilatometer. The study on the two or more dissimilar metals or alloys can be made with the newly developed thermal expansion characteristics. Moderate level of research has been done using existing studies. However, new researchers could use this as reference in future.

REFERENCES

[1] Rabin B, Swank W and Wright R 2013 Thermophysical Properties of Alloy 617 from 25 C to 1000 C Nuclear Engineering and Design 262 72-80
[2] Salimon A, Stepashkin A, Tcherdyntsev V, Olifirov L, Klyueva M and Kaloshkin S 2017 Towards the growth of single quasicrystalline grains in Al-Cu-(Fe, Cr) alloys after mechanical alloying and subsequent high temperature heating Journal of Alloys and Compounds 720 95-104
[3] Abe S, Sasaki F, Oonishi T, Inoue D, Yoshida J, Takahashi D, Tsujii H, Suzuki H and Matsumoto K 2012 A compact capacitive dilatometer for thermal expansion and magnetostriction measurements at millikelvin temperatures Cryogenics 52 452-6
[4] Raju S, Sivasubramanian K, Divakar R, Panneerselvam G, Banerjee A, Mohandas E and Antony M 2004 Thermal expansion studies on Inconel-600® by high temperature X-ray diffraction Journal of nuclear materials 325 18-25
[5] Choo H, Lee W, Hong S-J and Lee C 2016 Application of the dilatometer test for estimating undrained shear strength of Busan New Port clay Ocean Engineering 115 39-47
[6] Manohar R, Gupta M and Shukla J 2000 Phase transition studies of some cholesteric liquid crystals and their mixtures using dielectric, optical transmittance and density measurement techniques Journal of Physics and Chemistry of Solids 61 1465-73
[7] Apel M, Benke S and Steinbach I 2009 Virtual dilatometer curves and effective Young’s modulus of a 3D multiphase structure calculated by the phase-field method Computational materials science 45 589-92
[8] Ghanem H, Zollinger D, Lytton R and Ghanem N 2012 Determining ASR characteristics using dilatometer method Construction and building materials 36 1008-15
[9] Rafikov S, Leplyanin G, Varisova E, Naletova G and Kazakov V 1979 Control by complex formation of the inhibiting effect of anthracene in free radical polymerization Polymer Science USSR 21 2170-6

[10] Sunder V S and Umarji A 1995 Thermal expansion studies in the lead iron niobiate—Lead titanate system Materials research bulletin 30 427-34

[11] Srivastava A K, Bhattacharjee D, Jha G, Gope N and Singh S 2007 Microstructural and mechanical characterization of C–Mn–Al–Si cold-rolled TRIP-aided steel Materials Science and Engineering: A 445 549-57

[12] Kaiser D, de Graaff B, Dietrich S and Schulze V 2016 A novel procedure to account for high temperature gradients in an induction dilatometer sample during rapid heating Thermochimica Acta 646 8-15

[13] Manoj N, Jain D, Gautam J, Thomas K, Sudarsan V, Pillai C, Vatsa R and Tyagi A 2016 A simple, reliable and cost-effective, high temperature dilatometer for bulk thermal expansion studies on solids Measurement 92 318-25

[14] Yu H, Adams R and Da Silva L F 2013 Development of a dilatometer and measurement of the shrinkage behaviour of adhesives during cure International Journal of Adhesion and Adhesives 47 26-34

[15] Kaur K and Pandey O 2010 Microstructural characteristics of spray formed zircon sand reinforced LM13 composite Journal of alloys and compounds 503 410-5

[16] Chen F-W, Xu G, Zhang X-Y, Zhou K-C and Cui Y 2017 Effect of α morphology on the diffusional β→α transformation in Ti–55531 during continuous heating: Dissection by dilatometer test, microstructure observation and calculation Journal of Alloys and Compounds 702 352-65

[17] Yin S-b, Sun X-j, Liu Q-y and Zhang Z-b 2010 Influence of deformation on transformation of low-carbon and high Nb-containing steel during continuous cooling Journal of Iron and Steel Research International 17 7

[18] Mazur A V and Gasik M M 2009 Thermal expansion of silicon at temperatures up to 1100 C Journal of materials processing technology 209 723-7

[19] Özgün Ö, Gülsoy H Ö, Yilmaz R and Findik F 2013 Microstructural and mechanical characterization of injection molded 718 superalloy powders Journal of alloys and compounds 576 140-53

[20] Han W, Li Y, Chen G and Yang Q 2017 Effect of sintering additive composition on microstructure and mechanical properties of silicon nitride Materials Science and Engineering: A 700 19-24

[21] Unruan M, Prasatkhetragarn A, Laosiritaworn Y, Ananta S, Khamman O, Yimnirun R, Guo R and Bhalla A 2010 Thermal expansion behavior and estimated total polarizations of lead zirconate titanate—lead nickel niobate ceramics Materials Letters 64 1960-3

[22] Zi A and Palkowski H 2010 Direct strip casting and hot rolling of an AZ31 magnesium alloy Materials Science and Engineering: A 528 559-65

[23] Kivitz E, Palm B, Heinrich J, Blumm J and Kolb G 2009 Reduction of the porcelain firing temperature by preparation of the raw materials Journal of the European Ceramic Society 29 2691-6

[24] Li S, Deng L, Wu X and Wang H 2010 Low-frequency internal friction investigating of the carbide precipitation in solid solution during tempering in high alloyed martensitic steel Materials Science and Engineering: A 527 6899-903

[25] Kirby D, Chang D, Stratton F and Zinck J 2009 A differential capacitive thin film hydrogen sensor Sensors and Actuators B: Chemical 141 424-30
[26] Mitchell J, Freibert F, Schwartz D and Bange M 2009 Unconventional δ-phase stabilization in Pu–Ga alloys Journal of nuclear materials 385 95-8

[27] Takeichi Y, Nishiyama I and Yamada N 2006 Performance of dilatometer for determining absolute CTE of EUVL LTEMs Microelectronic engineering 83 1617-20