

Synopsis of the thesis entitled

**THERMODYNAMIC INVESTIGATIONS OF PHASE
TRANSFORMATIONS IN AMORPHOUS ALLOYS AND
POLYMERS**

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By

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THERMODYNAMIC INVESTIGATIONS OF PHASE TRANSFORMATIONS IN AMORPHOUS ALLOYS AND POLYMERS

Plan of the Thesis:

The following discussion gives the chapter-wise account of the work to be presented in the thesis.

Chapter I: This chapter deals with the general information about the amorphous materials, polymers and bulk metallic glasses. Their history, properties, applications etc. are discussed in this chapter.

Chapter II: Experimental Techniques are discussed in this chapter. Differential Scanning Calorimetry (DSC) and Modulated Differential Scanning Calorimetry (MDSC) are studied

Chapter III: This chapter provides the theoretical account of the thermodynamic properties i.e. Gibbs free energy difference, Entropy difference and Enthalpy difference of liquid metals and various bulk metallic glasses.

Chapter IV: This chapter provides the study of glass forming ability (GFA) of different amorphous alloys. Firstly, different GFA criteria for binary systems are studied

Chapter V: The study of crystallization kinetics of metallic glasses is discussed in this chapter. The experiments are carried out using Differential Scanning Calorimetry (DSC) for different multi-component metallic glasses. The data obtained from DSC are analyzed using different iso-conversional and iso-kinetic methods.

Chapter VI: The knowledge gained through this work and scopes for the future work are summarized in this chapter.

Chapter I Introduction

Matter can exist in three states: Solid, liquid and gaseous. The major difference between the three states lies in atomic arrangement of their constituent atoms. Solids can be classified as crystalline or amorphous. Crystalline solids are those materials which have periodic arrangement of their constituent atoms or molecules or ions and have a definite external geometrical form. On the other hand, amorphous solids are those materials which does not have a periodic arrangement of atoms and there is no long range order of the position of the atoms.

Glass is any material that can be cooled from a liquid to a solid without crystallizing. All kinds of materials can form glasses irrespective of their physical and chemical properties with the rapid solidification technique. The thermodynamic behavior is important for understanding the viscosity, glass forming abilities, nucleation and growth kinetics etc of the undercooled melt

Metals and their alloys are generally crystalline in nature and possess a long range order of the atom, with translational periodicity. Most of the metals crystallize when they are cooled, arranging their atoms into a highly regular spatial pattern called a lattice. But if crystallization is avoided then atoms settle into a nearly random arrangement and the resulting form is a metallic glass. Amorphous alloys also known as “**Metallic glasses**” are non-crystalline and random in nature.

But about a half century ago, materials scientists learned how to make glassy metals by cooling a metallic liquid so quickly that the internal atomic configurations froze before the atoms had a chance to arrange themselves into a lattice pattern. In 1960, first amorphous metallic glass material was produced by Duwez and co-workers when they synthesized Au-Si ribbons by quenching at 10^6 K/s. In the mid 80's, certain alloy systems were discovered that required far lower cooling rates ($\sim 10^2$ K/s) to achieve amorphous structure. This allowed fabrication of shapes of larger dimension samples of these amorphous alloys, the dimensions being in the order of millimeters (mm) instead of microns (μ). These alloys were known as ‘Bulk Metallic Glasses’ and have generated interest because of their potential applications. These metastable materials possess excellent properties such as high strength, high hardness, exhibit good corrosion resistance and attractive soft magnetic behavior [1].

The study of the thermally-activated phase transformations is of great significance in the field of materials science as the properties of materials change due to the change in the composition and/or microstructure. The properties of fully or partly crystalline materials are usually different from their amorphous counterparts. The bulk properties of the material are the manifestation of the atomic interactions and dynamics at the microscopic level. Since the amorphous state is thermodynamically unstable, the thermal stability of amorphous alloys is crucial from the application viewpoint. This makes the *thermo-analytical* study of the amorphous alloys an important field of research. The study of crystallization kinetics and kinetics of glass transition of amorphous alloys using thermo-analytical technique i.e. Differential Scanning Calorimetry (DSC) provides very useful information about the thermal stability [2].

Polymer molecules segments can exist in two specific physical structures. They can exist in either crystalline form or amorphous form. Crystalline polymers are possible in such conditions if there is a regular chemical structure (e.g., homopolymers or alternating copolymers), and the chains possess a highly ordered arrangement of their segments. Symmetrical polymer chains are never 100% and crystallinity in polymers is due to symmetrical polymer chains. Thus, polymers form mixtures of small crystals and amorphous material. These semi-crystalline polymers have a kind of typical liquifiction pathway, having their solid state until they reach their melting point at T_m . So it is interesting to study the thermodynamic behavior of polymers.

BMGs have an interesting combination of properties. They exhibit very high strength, large elastic elongation limit, very high hardness, excellent corrosion resistance, and a good combination of soft magnetic properties. There are existing and potential applications of BMG which are discussed in this chapter of thesis.

Chapter II Experimental Techniques

The heat into or out of a sample is measured by calorimeter. The general instrumentation for the thermal analysis primarily includes:

- i. A sample holder, where the sample is kept
- ii. Sensors for detecting the required properties with respect to temperature
- iii. A closed chamber where the sample and sensors can be placed and the experimental parameters can be controlled according to the requirements.
- iv. A computer for adjusting the experimental parameters, collecting data and performing further calculations.

Differential Scanning Calorimetry (DSC) can be used to measure a number of characteristic properties of a sample. Using this technique it is possible to observe fusion and crystallization events as well as glass transition temperatures T_g . DSC can also be used to study oxidation, as well as other chemical reactions. Glass transitions may occur as the temperature of an amorphous solid is increased. These transitions appear as a step in the baseline of the recorded DSC signal. This is due to the sample undergoing a change in heat capacity; no formal phase change occurs. The ability to determine transition temperatures and enthalpies makes DSC a valuable tool in producing phase diagrams for various chemical systems.

DSC is used widely for examining polymeric materials to determine their thermal transitions. The observed thermal transitions can be utilized to compare materials, although the transitions do not uniquely identify composition. The composition of unknown materials may be completed using complementary techniques such as IR spectroscopy. Melting points and glass transition temperatures for most polymers are available from standard compilations, and the method can show polymer degradation by the lowering of the expected melting point, T_m , for example. T_m depends on the molecular weight of the polymer and thermal history, so lower grades may have lower melting points than expected. The percent crystalline content of a polymer can be estimated from the crystallization/melting peaks of the DSC graph as reference heats of fusion can be found in the literature.

A differential calorimeter, measures the heat of sample relative to a reference. Differential Scanning Calorimetry (DSC) is a technique in which difference in heat flow to a sample and to reference pan is monitored against time or temperature. The

temperature of sample and reference are made identical by varying the power input to the two surfaces and heats the sample with a linear temperature ramp. The energy required to do this is the enthalpy or heat capacity changes in the sample relative to reference. In the endothermic reactions, heat flows into the sample whereas in exothermic reactions heat flows out of the sample. DSC is a method of thermal analysis that is widely used to study thermal transitions, i.e., solid-solid transitions as well as solid-liquid and various other transitions and reactions. By using thermal analysis it is possible to understand what is happening in a material even if there is no visual evidence that a change has occurred. In a controlled atmosphere DSC measures the temperatures and heat flows associated with transitions in materials as a function of time and temperature. It also provides quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes of materials during physical transitions that are caused by phase changes, melting, glass transitions, crystallization, oxidation, and other heat related changes. This information helps the scientist or engineer identify processing and end-use performance. The DSC instrument works in conjunction with a controller and associated software to allow for data acquisition and analysis. Using DSC, one can better understand phase transitions and reactions in materials, and how they contribute to the properties and characteristics of the material. DSC is an efficient and powerful tool for quick determination of the specific heat and enthalpy of change accompanying the primary or secondary phase transition of substance. The data of crystallized fraction with temperature or time can be procured from DSC runs at different heating rates and the crystallization process in metallic glass can be studied by both isothermal and non isothermal conditions [3-5].

Types of DSC

There are mainly two types of DSC: 1) Power compensated and 2) Heat flux. The information obtained from both DSC is same but their instrumentation is different

Chapter III Thermodynamic Properties of metallic glasses and polymers

The thermodynamic properties of the undercooled melt is important for understanding the viscosity, glass forming abilities, nucleation and growth kinetics

etc. The temperature dependence of the Gibbs free energy difference (ΔG), enthalpy difference (ΔH) and entropy difference (ΔS) between the undercooled melt and the corresponding equilibrium solid have been analyzed for **metallic glasses** and glass forming polymeric materials

The estimation of the Gibbs free energy change (ΔG) is often critically important when used in the analysis of nucleation phenomena because the nucleation frequency has an exponential dependence on ΔG . Varieties of expressions for ΔG are available in the literature.[6-15]. These expressions, on one hand, depend on some kind of assumption for the temperature dependence of the heat capacity. On the other hand, the derivation of expression for ΔG is based on the assumption that under cooling ΔT ($= T_m - T$) is small. This limits the validity of the different proposed expressions for ΔG over small temperature interval. But, the BMG forming alloys possess large undercooled regime. Therefore, the need for an expression of ΔG applicable over a wide temperature range was strongly felt.

There are basically three different kinds of systems depending on the variation of specific heat difference (ΔC_p) with temperature. It is seen that in the case of magnetic liquid melts like Cobalt and Nickel the ΔC_p decreases with increasing undercooling while in case of most of glass forming metallic glasses the ΔC_p values remain nearly constant with undercooling. In few bulk metallic glasses the ΔC_p increases with temperature. Considering, the different variations of ΔC_p , two different expressions for ΔG derived are given in *Chapter 2* of the thesis.

The difference in Gibbs free energy between the liquid and crystalline phases is given by

$$\Delta G = \Delta H - T\Delta S \quad (1)$$

Where

$$\Delta H = \Delta H_m - \int_T^{T_m} \Delta C_p dT \quad (2)$$

$$\text{and} \quad \Delta S = \Delta S_m - \int_T^{T_m} \Delta C_p \frac{dT}{T} \quad (3)$$

$$\text{with} \quad \Delta S_m = \frac{\Delta H_m}{T_m} \quad (4)$$

Experimental ΔG can be calculated using equations 1-3. Assuming ΔC_p to be constant, substituting ΔH and ΔS from Eq. (2) and (3) in Eq.(1) provides

$$\Delta G = \frac{\Delta H_m}{T_m} \Delta T + \Delta C_p \left[T \ln \frac{T_m}{T} - \Delta T \right] \quad (5)$$

Treating ΔC_p to be constant in the undercooled region and taking $\Delta S = 0$, one can easily get at $T = T_K$,

$$\Delta C_p = \alpha \frac{\Delta H_m}{T_m} \quad (6)$$

$$\text{with } \alpha = \frac{1}{\ln T_m / T_K}$$

Substituting this expression for constant ΔC_p in Eq.(5), the new expression for ΔG for multicomponent BMG forming alloys with large undercooled region comes out to be

$$\Delta G = \frac{\Delta H_m}{T_m} \left[\alpha T \ln \frac{T_m}{T} + \Delta T (1 - \alpha) \right] \quad (7)$$

Except for the assumption related to ΔC_p , it does not involve any other approximation.

Although the experimental ΔC_p 's are far from being constant, the one-parameter formula for ΔG (involving a constant ΔC_p) fits very well with the experimental data for most of the bulk amorphous alloys. In the present work, α has been derived using the following expression obtained from Eq. (6) taking the constant value of $\Delta C_p = \Delta C_p^m$ at melting point

$$\alpha = \frac{\Delta C_p^m T_m}{\Delta H_m} \quad (8)$$

However, for such systems, in which specific heat increases considerably with undercooling, ΔC_p at any temperature $T_K < T < T_m$ in the undercooled region can be expressed as

$$\Delta C_p = \frac{\Delta C_p^m T_m}{T} \quad (9)$$

ΔC_p^m being the specific heat difference at the melting point. Substituting ΔC_p from Eq. (9) in Eqs. (2) & (3) and using Eq. (1), the ΔG values for such systems can be represented by the following equation

$$\Delta G = \frac{\Delta H_m \Delta T}{T_m} - \Delta C_p^m T_m \left[\ln \frac{T_m}{T} - \frac{\Delta T}{T_m} \right] \quad (10)$$

The Gibbs free energy difference is calculated using these ΔG expressions for various glass forming systems like $\text{Cu}_{47}\text{Ti}_{34}\text{Zr}_{11}\text{Ni}_8$, $\text{Pd}_{40}\text{Cu}_{30}\text{Ni}_{10}\text{P}_{20}$ etc. and for the glass forming polymeric melt ; polyamid-6 (PA-6), polypropylene oxide (PPO), polyethylene (PE), polytetramethylene oxide (PTMO), polyethylene oxide (PEO), polypropylene (PP) polybutadiene (PB), polyethylene terephthalate (PET) and two simple organic liquids: tri- α -naphthyl benzene(tri- α -NB) phenyl salicylate (salol) The detailed formulation and the results for ΔG along with the comparison with the experimental results is given in **Chapter 2**.

Using the above expressions, Gibbs free energy ΔG , Entropy difference ΔS , and Enthalpy difference ΔH are calculated for various glass forming systems like polyethylene (PE), polytetramethylene oxide (PTMO) polyamid-6 (PA-6), polypropylene oxide (PPO) and two simple organic liquids: *o*-terphenyl (*o*-ter), tri- α -naphthyl benzene (tri- α -NB).

The detailed formulation and the results for thermodynamic properties along with the comparison with the experimental results are given in this chapter of thesis.

Chapter IV Glass Forming Ability

The glass forming ability (GFA) of amorphous material is very important from both theoretical and practical point of view. The glass forming ability GFA is considered in three different ways namely kinetic, structural and thermodynamic point of view. From the kinetic point of view, viscosity is closely related to the reduced glass transition temperature ($T_{rg}=T_g/T_l$) where T_g is glass transition temperature and T_l is liquidus temperature. Higher T_{rg} leads to the higher GFA .[16]. The T_x/T_l ratio is also an indicator of the GFA where T_x is onset crystallization temperature and T_l is liquidus temperature. The large T_x/T_l ratio indicates a lower R_c and therefore the higher GFA[17]. R_c is a critical cooling rate, which is the

minimum cooling rate necessary for keeping the melt amorphous without crystallization. Actually R_c is a key parameter to evaluate the GFA of a melt but it is difficult to measure precisely. So we have used simple and reliable criteria for the GFA. From the structural point of view, higher T_g/T_m ratio and higher $\Delta T_{xg}=T_x-T_g$ is required for the higher GFA. These are the parameters to gauge the glass stability.

Thermodynamically, ΔG is a key parameter for predicting the GFA. Low value of ΔG means the driving force for crystallization is low and glass formation becomes easy. In the relation $\Delta G = \Delta H_f - T\Delta S_f$ for Gibbs free energy, ΔH_f and ΔS_f are enthalpy of fusion and entropy of fusion. In multicomponent systems, the number of microscopic states is large leading to the increase in ΔS_f , leads to a decrease in ΔG . Small value of ΔG is favourable for the higher GFA. The expressions are proposed to calculate ΔG , ΔS and ΔH using linear and hyperbolic variations of ΔC_p in the undercooled region for bulk metallic glasses and ZBLAN glass. Theoretical results show excellent agreement with experimental values. Different GFA criteria are also evaluated for bulk metallic glasses and ZBLAN glass is investigated.

Gibbs free energy difference, ΔG as a function of temperature, using Heena-1 et al., Heena-2 et al Lad-1 *et al.* Lad-2, Battezzati et al expression and other GFA parameters are also calculated for different multi component metallic alloys.

Chapter V Crystallization Kinetics of Metallic Glasses

In this chapter, the kinetics of crystallization of Zr-based metallic glasses has been investigated using DSC technique. The as-quenched samples were heated in DSC (DSC-50, Shimadzu, Japan) at four linear rates from room temperature to 800K in air. The DSC scans were recorded by a thermal analyzer (TA-50 WSI, Shimadzu, Japan) interfaced to a computer. The thermograms for both the samples show three-step crystallization.

Kinetic parameters are obtained from non-isothermal rate laws by both iso-kinetic and iso-conversional methods.

Iso-conversional Methods

Iso-conversional Methods calculate E_a values at progressive degrees of conversion without modelistic assumptions.

Linear Iso-conversional Methods

a) *Integral Iso-conversional methods*

The Linear integral iso-conversional methods are based on the integral form of rate law given by

$$g(\alpha) = \frac{A}{\beta} \int_0^T \left[\exp\left(-\frac{E}{RT}\right) \right] dT \equiv \frac{AE}{\beta R} p(x) \quad (13)$$

where $x = E/RT$ and $p(x)$ is the temperature integral which has no analytical solution. Hence, different researchers have used different approximations to calculate this temperature integral.

The approximation for $p(x)$ given by Coats and Redfern

$$p(x) \cong \frac{\exp(-x)}{x^2} \quad \text{for } 20 < x < 50 \quad (14)$$

is used by KAS in Eq. (13) and obtained the equation as

$$\ln\left(\frac{\beta}{T_\alpha^2}\right) = \ln\left(\frac{AR}{E_\alpha g(\alpha)}\right) - \frac{E_\alpha}{RT_\alpha} \quad (15)$$

The general equation for the special cases of KAS method when $T_\alpha = T_P$ is written as

$$\ln\left(\frac{\beta}{T_p^s}\right) = -Z \frac{E}{RT_p} + \text{const.} \quad (16)$$

When (1) $s = 2$ and $Z = 1$ Eq. (16) reduces to Kissinger equation

(2) $s = 1$ and $Z = 1$ Eq. (16) reduces to Boswell equation

(3) $s = 0$ and $Z = 1.052$, Eq. (16) converts to Ozawa equation.

Another special case of KAS is suggested by Augis and Bennett as

$$\ln\left(\frac{\beta}{(T_p - T_o)}\right) = -\frac{E}{RT_p} + \ln A \quad (17)$$

where T_p and T_o are the peak and onset temperatures respectively.

To estimate the value of Avrami exponent, n also, Augis and Bennett have derived an equation as

$$n = \frac{2.5 T_p^2}{\Delta T \frac{E}{R}} \quad (18)$$

where ΔT is the full width at half maximum of the DSC curves.

Ozawa - Flynn and Wall derived the following relation

$$\ln \beta = \ln \frac{AE_\alpha}{Rg(\alpha)} - 5.331 - 1.052 \frac{E_\alpha}{RT_\alpha} \quad (19)$$

By using the Doyle approximation given by

$$\ln p(x) = -5.331 - 1.052x \text{ in Eq. (14)}$$

At $T_\alpha = T_p$, the above Eq.(19) reduces to Ozawa equation.

b) *Differential Isoconversional methods*

Friedman derived a linear differential isoconversional expression as

$$\ln \left(\frac{d\alpha}{dt} \right)_\alpha = \ln (A f(\alpha)) - \frac{E_\alpha}{RT_\alpha} \quad (20)$$

A method suggested by Gao and Wang method uses the following expressions to determine the activation energy, frequency factor and Avrami exponent n.

$$\ln \left[\beta \left(\frac{d\alpha}{dT} \right)_p \right] = - \frac{E}{RT_p} + \text{const}, \quad \text{where } \beta = dT/dt \quad (21)$$

$$K_p = \frac{\beta E}{RT_p^2} \quad (22)$$

$$\text{where } K_p = A \exp \left(\frac{-E}{RT_p} \right)$$

$$n = \frac{\left(\frac{d\alpha}{dt} \right)_p}{0.37 K_p} \quad (23)$$

This method is a special case of Friedman method as Eq.(19) is obtained by using only the peak temperature, T_p instead of T_α .

Isokinetic Methods

Matusita and Sakka method

Matusita and Sakka suggested the following equation specifically for the non-isothermal data

$$\ln[-\ln(1-\alpha)] = -n \ln \beta - \frac{mE_c}{RT} + Const \quad (24)$$

Where m is an integer depends on the dimensionality of the crystal and the Avrami exponent n depends on the nucleation process.

Modified Kissinger method

The modified Kissinger equation given below can be utilized to derive the activation energy (E).

$$\ln\left(\frac{\beta^n}{T_p^2}\right) = -\frac{mE}{RT_p} + Const \quad (25)$$

Where E is the activation energy for crystallization, T_p is the peak temperature, and R is the universal gas constant.

The different kinetic parameters are calculated using the above mentioned equations for both the systems. The results along with the detail calculations are discussed in this chapter of thesis.

Chapter VI Conclusion and Scope for the Future Work

The work to be presented in the thesis will give details of the thermodynamic properties of different glass forming systems and polymers.

Theoretical analysis includes the study of thermodynamic aspect of glass formation in metallic glasses along with the determination of their glass forming ability (GFA). The thermodynamic parameters i.e. Gibbs free energy difference (ΔG), entropy difference (ΔS) and enthalpy difference (ΔH) were calculated for an Au-based metallic alloy in its undercooled region. The calculations of ΔG was done on the basis of various expressions available in literature depending upon different variations of specific heat difference (ΔC_p) with temperature. The theoretical formulation gives improvement over existing analytical models and provides better understanding of the thermodynamical properties of the amorphous alloys and polymers. Thermodynamically, the Gibbs free energy difference, ΔG between the under cooled

liquid and the corresponding crystalline state is driving force for crystallization. As a consequence, it is a good indicator for glass forming ability of metallic glasses. The activation energy is a good indicator of thermal stability. Crystallization kinetics and kinetics of glass transition are the ways to determine the activation energy and other kinetic parameters. Thus, we can predict glass forming ability and thermal stability of glassy alloy and ZBLAN glass.

The experimental investigations provide insight of crystallization kinetics of Zr based Zr₅₂Cu₁₈Ni₁₄Al₁₀Ti₆ amorphous alloy. Crystallization kinetics is the way to determine the activation energy and other kinetic parameters of the metallic glass. Thus, we can predict glass forming ability and thermal stability of glassy alloy. Crystallization of metallic glasses involves nucleation and growth processes. DSC technique is used to study crystallization kinetics of Zr₅₂Cu₁₈Ni₁₄Al₁₀Ti₆ metallic glass and glass forming polymer melts. The knowledge gained through the work and the issues arising out of this are summarized in the *Chapter 6* of the thesis.

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(Prapti B. Pandya)

Signature of Candidate

Endorsement of the Supervisor:

Synopsis is approved by me

(Arun Pratap)

Research Guide

Professor of Condensed Matter Physics

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Dean

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List of Publications based on the research work:

1. *Thermodynamics of glass forming polymeric melts*
Prapti B. Pandya, Ashmi T. Patel, and Arun Pratap
AIP Conf. Proc. 1536, 655-656 (2013)
2. *Thermodynamic Behaviour of Glass Forming Polymeric Melts*
Prapti B. Pandya, Ashmi T. Patel, Arun Pratap
[Submitted to Polymer Bulletin]
3. *Glass Forming Ability of ZBLAN Glass Forming System*
Prapti B. Pandya, Arun Pratap
[Submitted to International Journal of Thermophysics]

Paper Presented at Conferences:

1. Thermodynamics of Glass Forming Polymeric Melts

Prapti B. Pandya, Ashmi T. Patel, and Arun Pratap
XXVI Gujarat Science Congress
February 2012, Vadodara, Gujarat
 2. Thermodynamic Parameters of glass forming polymeric melts
Prapti B. Pandya, Ashmi T. Patel, and Arun Pratap
National Conference on Current Trends in Material Research
(CTMR-2012) Jaipur, Rajasthan
 3. Thermodynamics of glass forming polymeric melts
Prapti B. Pandya, Ashmi T. Patel, and Arun Pratap
International Conference on Recent Trends in Applied Physics and Material Science
(RAM-2013) Bikaner, Rajasthan
-

FROM: _____

Phone No. _____

Date: _____

To

The Registrar (Examination),

The Maharaja Sayajirao University of Baroda

Vadodara

Subject: To submit _____

Through: The Guiding Teacher Department of _____

The Head, Department of _____

The Dean Faculty of Technology and Engineering

Sir,

I have registered for Ph.D. in _____ my

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on _____ in the faculty office. I am submitting herewith FOUR Copies of my Thesis
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submitted as per the university rules.

Thanking you,

Yours Sincerely,

(Name of the candidate & signature)

Encl: as above