Ceramics Materials Manufacturing

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2017

Green Body Drying

This section discusses the kinetics of drying for various conditions of temperature and solvent partial pressure in the atmosphere.

INTRODUCTION

To remove the solvent used to suspend the ceramic powder for green body fabrication, the green body is heated or placed in an atmosphere where the solvent evaporates.

Depending on the type of ceramic powder used and the green body fabrication process different solvents are used.

LUBRICANT ADDITION

Solvents are typically organics or water.

Organic solvents are chosen to easily wet the ceramic powder surface and easily evaporate during drying.

This means that a low enthalpy of vaporization and a reasonably high vapor pressure is important for a solvent to be easily evaporated.

The weight loss during drying is shown in Figure .1.

This figure shows a constant rate period where the surface of the green body is always wet by the flow of liquid to the surface.?

This flow emanates from the rearrangement of particles in the green body, which is caused by the compressive capillary pressure at the surface of the green body.

At some point, the particle network becomes rigid and no more shrinkage can take place.

LUBRICANT ADDITION

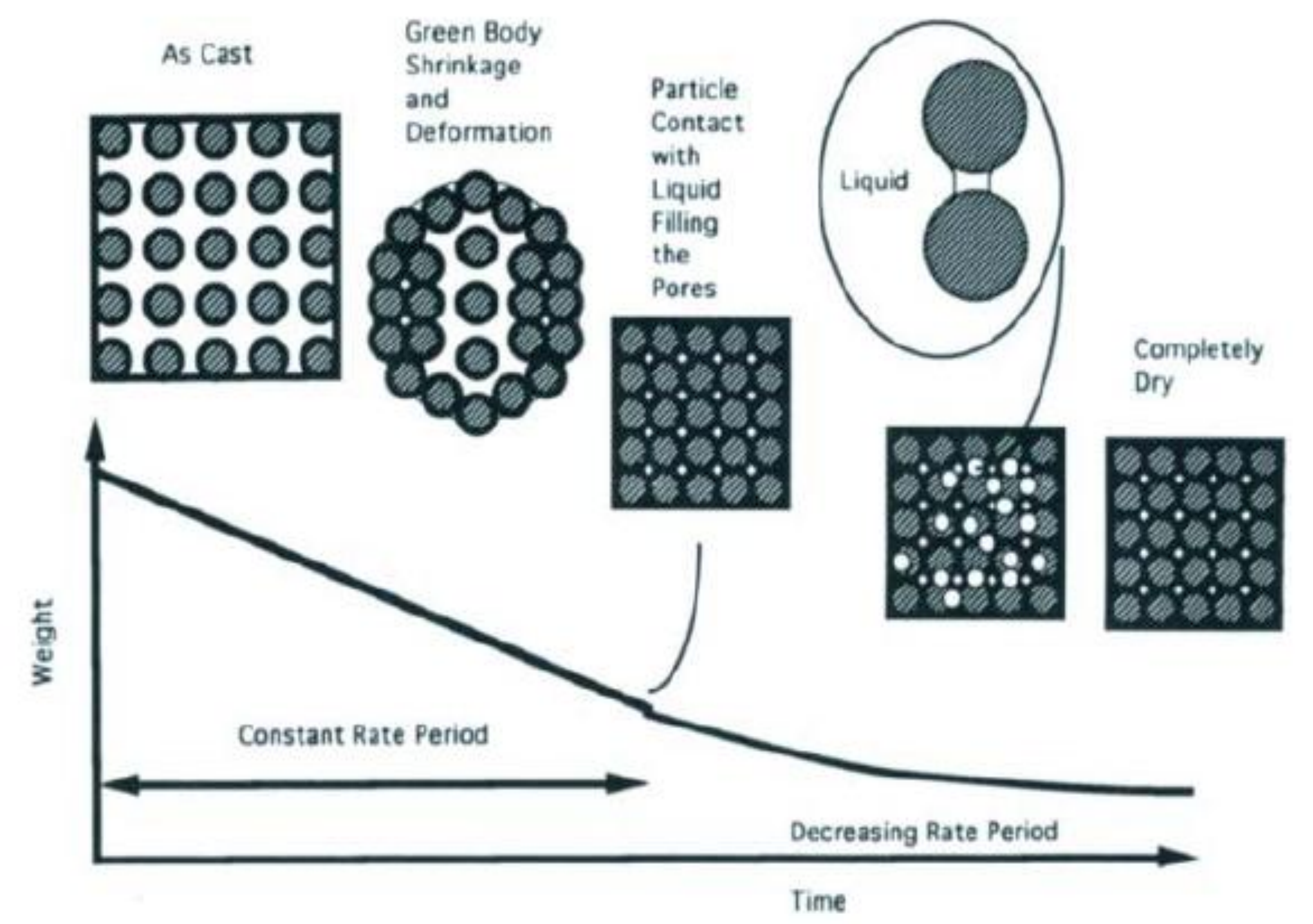


FIGURE .1 diagram of the drying of a ceramic green body showing the weight loss and shrinkage with time

LUBRICANT ADDITION

This critical point is referred to as the **leather hard point** in clay ceramics nomenclature.

With particulate ceramics, this **rigidity threshold** takes place at **high volume fraction where the particles come into contact**.

With **gels**, the particle network exists at the start of drying--shrinkage occurs as a result of **this compliant network stopping at the rigidity threshold of the network**. After this critical point, the liquid-vapor interface starts to recede into the pores.

At this stage, surface tension **driven flows** in the direction of the free surface attempt to keep a monolayer of solvent on the surface of all the ceramic powder.

Such capillary flow will continue as *long as there* is a **continuous pathway from the liquid front to the green body surface**.

LUBRICANT ADDITION

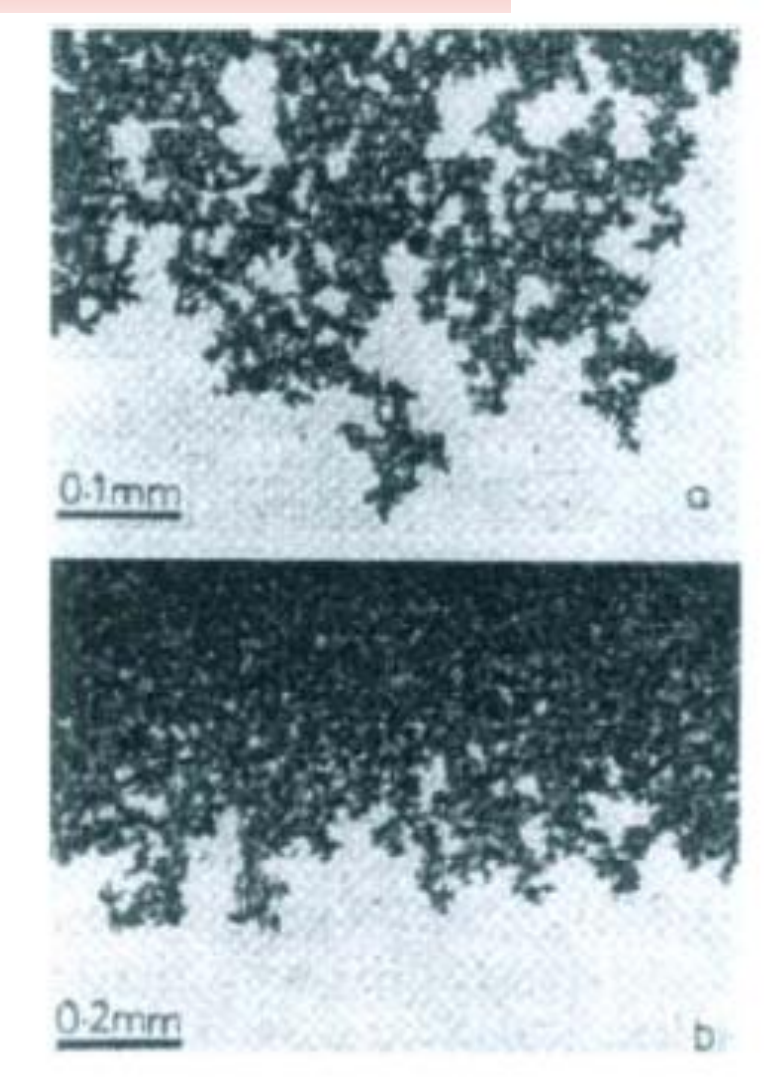
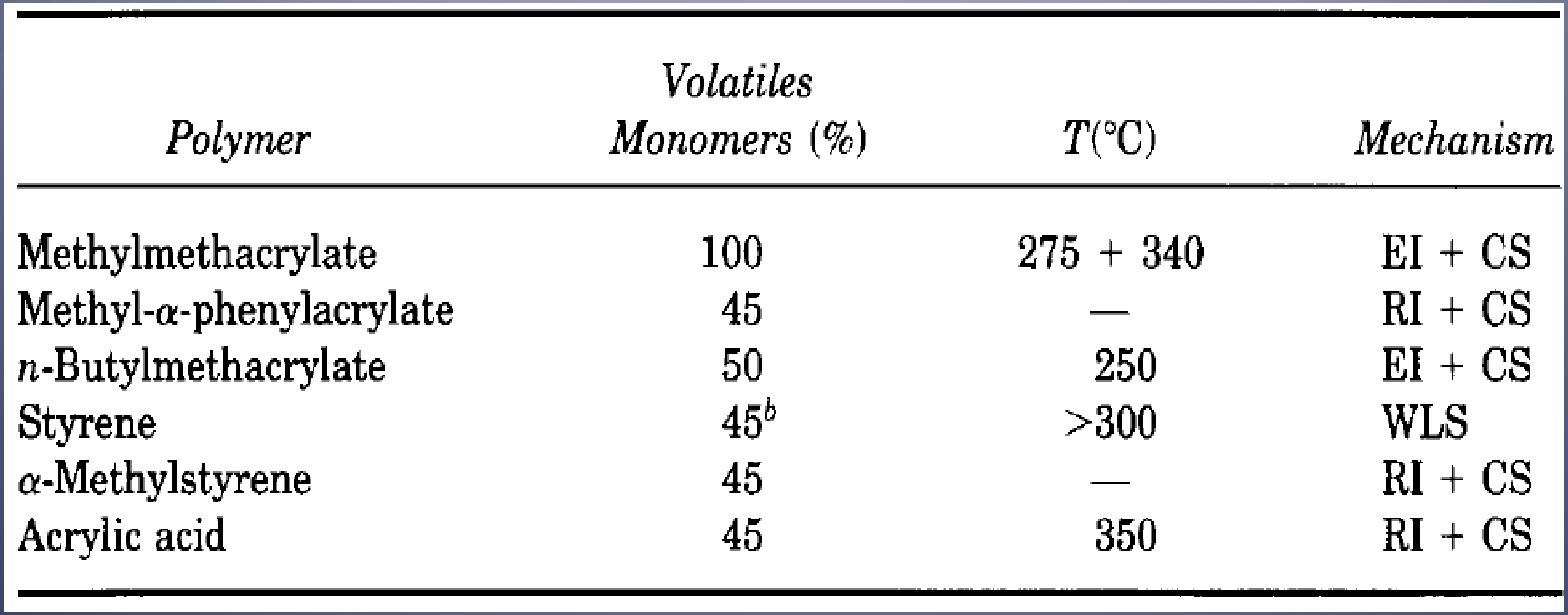


FIGURE 2 The drying front in a green body composed o

f mono sized 0.5 mm SiO_2 particles

LUBRICANT ADDITION

TABLE .1 List of Polymers That Depolymerize during Thermal Degradation



Polymer	*Volatiles Monomers (%)*	*T(°C)*	*Mechanism*
Methylmethacrylate	100	275 + 340	EI + CS
Methyl-α-phenylacrylate	45	—	RI + CS
n-Butylmethacrylate	50	250	EI + CS
Styrene	45[b]	>300	WLS
α-Methylstyrene	45	—	RI + CS
Acrylic acid	45	350	RI + CS

DRYING MECHANISM
(DRYING PROCESS)

1.Heat Transfer

The flux of heat, q, into a green body is given by the boundary layer heat transfer:

$$q = Q/A = h (T_B - T_S)$$

Where

Q is the quantity of heat transferred per unit time,

A is the surface area of the green body at temperature T_S exposed to the bulk gas at temperature T_B, and

h is the heat transfer coefficient which is a function of the gas flow rate around the green body.

DRYING MECHANISM
(DRYING PROCESS)

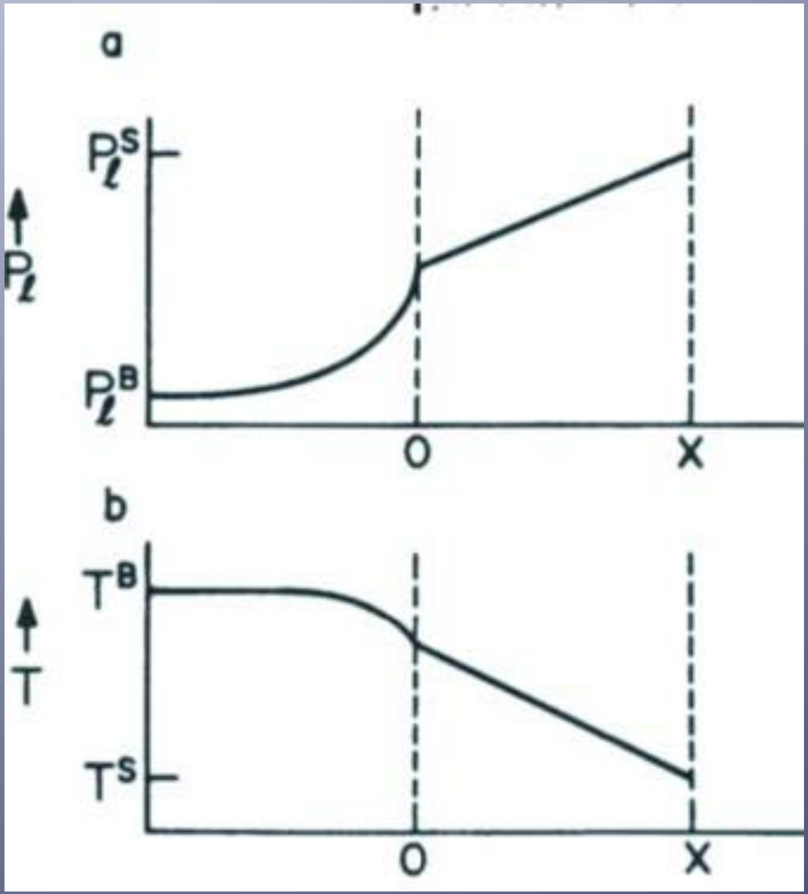


FIGURE .3 diagram of evaporation in a porous network (a) liquid partial pressure profile, (b) temperature profile.

DRYING MECHANISM
(DRYING PROCESS)

2. Mass Transfer

The mass flux, **j**, is related to the heat transfer flux, **q**, required to evaporate those molecules at the point of evaporization by the following expression:

$$q = \Delta H_{\mathbf{vap}} * j$$

Where $\Delta \mathbf{H}_{\mathbf{vap}}$ is the enthalpy of reaction.

This relation requires that the two differential equations for the two fluxes be linked for their simultaneous solution, the mass transfer flux **j**.

DRYING RATE

Once the drying front enters the green body, the **drying rate decreases** .

Because water evaporates on the material surface during the constant drying-rate period, the *drying rate **Rd*** is apparently **equal to** the *evaporation rate* from the free water surface

$$\theta_d = \int_{w_1}^{w_2} \frac{dw}{R_d}$$

The drying time q_d , drying rate R_d, water content w

STAGES OF DRYING

As shown in Figure . the drying process consists of the following
three stage :

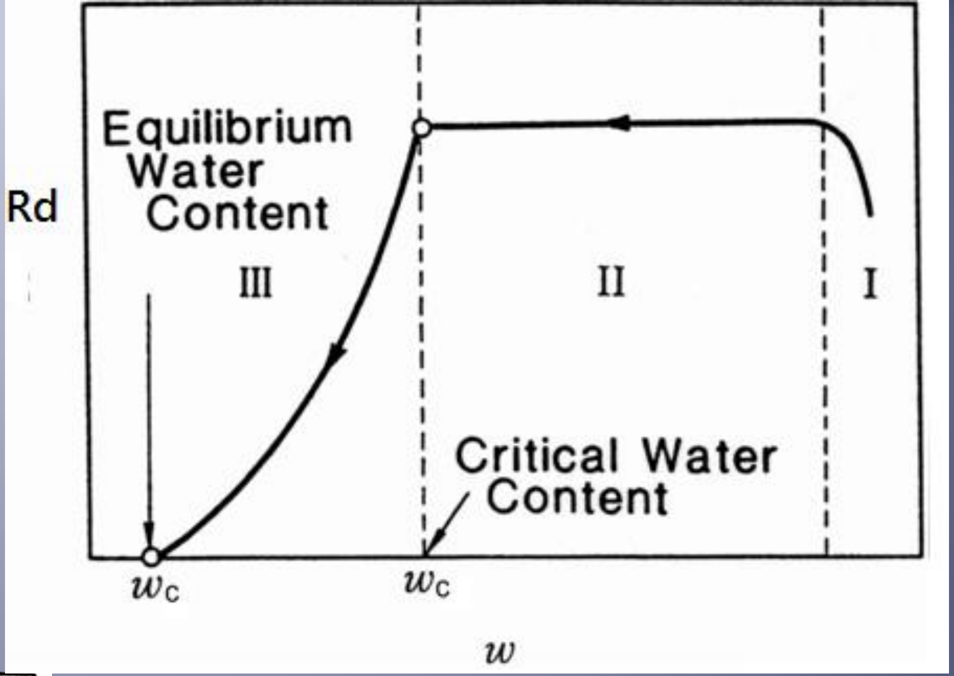
I) preheating period,

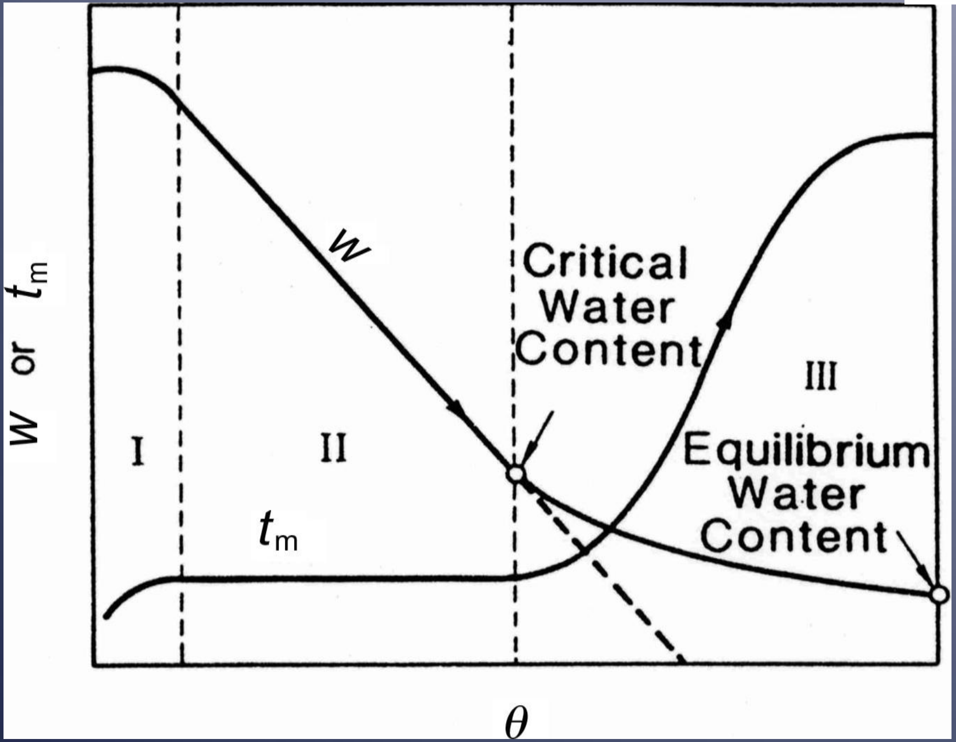
II) constant drying-rate period: free water exists on the surface of the material

III) decreasing drying rate period.

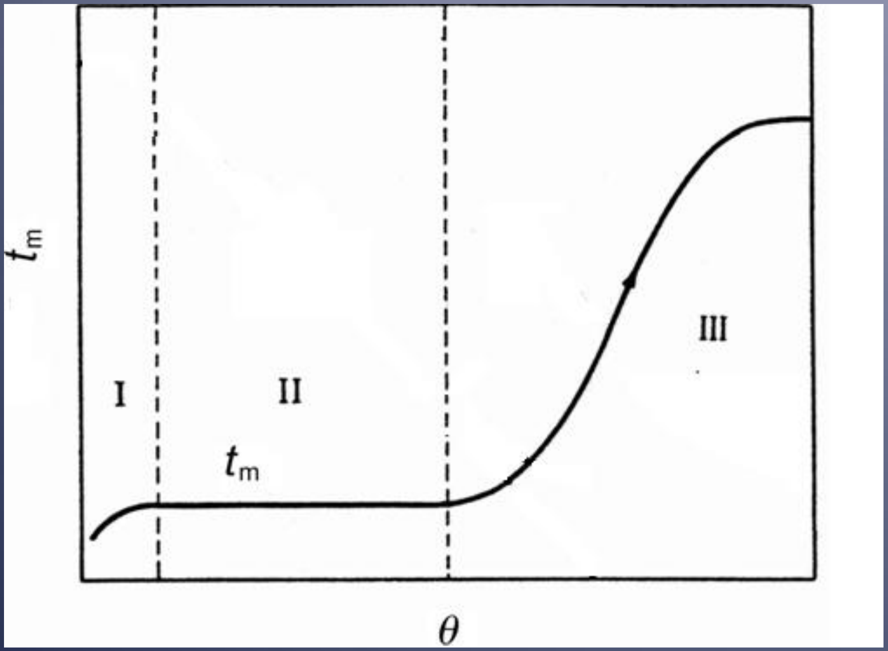
Figure shows the drying rate $\boldsymbol{R}_d$ in relation to the water content w.

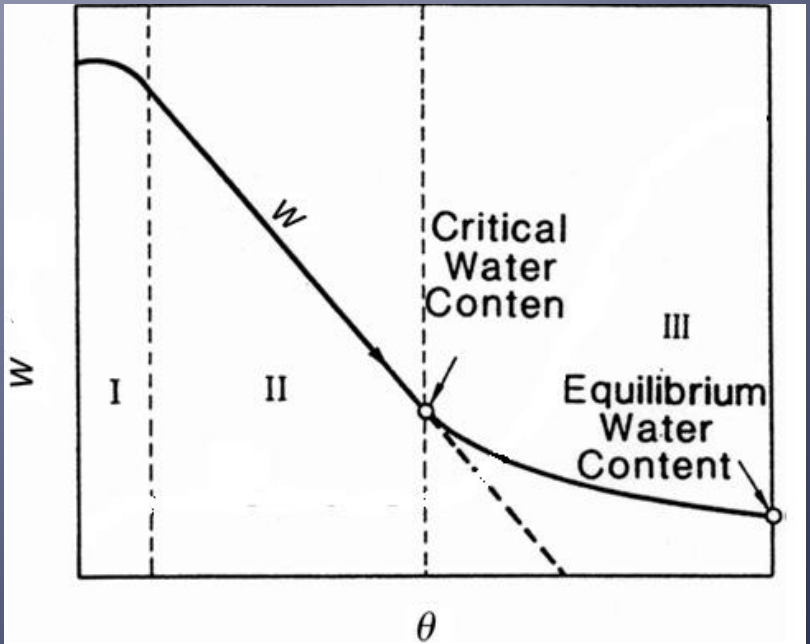
STAGES OF DRYING





STAGES OF DRYING





DRYING STRESSES

The local effective stress $\boldsymbol{\sigma_e}$ is given by the sum of the gas pressure, P_g, the capillary pressure, $\boldsymbol{P_c}$, and stress within the solid $\boldsymbol{\sigma_s}$**:**

$$\boldsymbol{\sigma_e = \sigma_s + P_g + P_c}$$

The net stress is given by the difference between the effective stress and the average stress:

$$\boldsymbol{\sigma = \sigma_e - \langle\sigma\rangle}$$

Where **<σ>** is the average stress integrated over the pore volume of the green body, ***Vp*** given by

$$\langle\sigma\rangle = \frac{1}{V_p}\int_0^{V_p} \sigma_e \, dV$$

The stress within the solid is often to be **constant** over the green body, thus it disappears from the net stress.

If, however, there is a temperature profile in the green body.

DRYING STRESSES

In the initial stage of drying, when the liquid fills the pores and there is a temperature profile in the green body, the thermal expansion of the liquid will add another stress, that can be substantial.

However, this liquid expansion stress is either

- not Important because the liquid filling the pores transfers heat relatively fast and eliminates thermal gradients .
- relieved by flow if the permeability of the particle network is high.

CRACKING, AVOIDANCE OF CRACKING

A small amount of polymer can produce an **enormous volume of gas**, which must be removed from the porous green body.

The flow of this gas in the porous network of the ceramic green body can create a <u>pressure build-up</u> which *puts* stress on the green body.

The stress induced by flow and temperature gradients in the green body , so that <u>binder fatigue conditions</u> can be selected to prevent **cracking** of the green body.

CRACKING, AVOIDANCE OF CRACKING

To remove the polymeric binder, the green body is heated often in an oxidizing atmosphere.

During this process the polymer **degrades** along many possible degradation pathways, which include

(1) Separation of the main chain,
(2) Reaction with side chains and sub-stituents,
(3) Cross-linking ending in carbon formation.