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FILM BOILING TRANSITION TEMPERATURE FOR TISSUE COOLED WITH LIQUID NITROGEN

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ABSTRACT

A series of tests were undertaken to determine the film boiling transition temperature (T_{DFB}) of in-vitro pigskin using liquid nitrogen as the coolant. No unique value was found; however for the horizontal minimum disturbance system $T_{DFB} \approx 243$ K and for irregular surface topology T_{DFB} ranged from 260 to 275 K. Lesions and surface particles were found to cause immediate transition followed by high cooling rates (> -80 K/sec). Lesions or particles can be used for controlled cooling in varying geometric patterns. Contouring and surface masking using plastics and metals can also be used to control cooling rates (approx. -.6 K/sec for a plastic spoon reservoir).

In-vivo experiments on fingers were in close agreement with the in-vitro tests on pigskin. No deep freezing was permitted.

INTRODUCTION

In cryobiology and cryosurgery, the preservation and destruction of tissue is accomplished by varying the cooling rate, the temperature level, and the duration at that level. Skin cancers and growths have been successfully treated by spraying liquid nitrogen on the defective tissue (1). Thousands have been treated since 1962 for involuntary movement disorders (parkinsonism, dystonia musculorum deformaus, intention tumor, and torticollis), using stereotoxic cryothalamectomy (2). A 2 mm cryogenic cannula is carefully positioned and destruction of the tissue is affected by lowering the probe temperature levels below 253 K for various time periods. Here the rate, level and period of cooling are critical. Gynecological disorders have been treated using cryogenic cannula techniques with a variety of freezing surface configurations (3).

A variety of techniques using cryogens are available for the preservation of whole blood and its constituents for periods of years (4). Here the type of cryoprotective compound used (glycerol, dimethyl sulfoxide or polyvinylpyrrolidone) as well as cooling rates are important to preservation. Cryogens have been used to preserve foodstuffs, such as fish, poultry, red meats, fruits and vegetables (mushrooms, tomato slices, peas, beans, berries, sliced peaches, etc.) (5 and 6).

Currently much effort is being devoted to whole organ preservation. The recent work of Lehr (7) indicates that the kidney may be able to survive freezing when microwave energy is used to revitalize the organ. Lehr's work points out the importance of acquiring procedures for the entire cycle (cooling-storage-warming) in organ preservation.

Such procedures are extremely important for the recovery of biological specimens from the planets such as Mars. The problem involves cultures which may be dormant and if improperly handled would remain forever extinct and beyond our means of detection of the life forms. Cooling rates prior to the lengthy interplanetary voyage, the storage temperature during transit, and of course the rate of revival, all have a direct influence on the survival of the culture. In some instances we may indeed be dealing with materials which are "dead" by definition, but the term "dead" may be just a catch all phrase to remind us of our inability to identify and revitalize the basic culture. To illustrate my point, properly frozen and preserved blood is "dead", and improperly revived to room temperature is as lethal to a human as poison, and is by definition not viable, i.e. "dead". However proper heating rates and cleansing to remove the cryoprotectant, even after years of proper storage, yields viable blood which the body can accept and use (4).

Other problems such as virus containment and disposal within the spacecraft are of concern to the space effort.

The above examples illustrate the usefulness of cryogens in specimen presentation or destruction. Although it is unnecessary to use a fluid which will ebullate, the extremely high heat transfer rates associated with boiling makes such a fluid very attractive. The rates of cooling as well as warming are critical to both specimen preservation and destruction. Herein we are concerned primarily with cooling rates. While the boiling curve (Fig. 1) is no panacea in defining heat transfer rates, it does serve to separate three classes of boiling: nucleate, transition, and film, according to heat flux and temperature difference. The identification of these regimes and the associated rates of heat transfer are very important to specimen preservation (or destruction). Herein we will be concerned with determining the point where stable film boiling terminates and transition boiling begins.

If a quantity of liquid is placed on a sufficiently hot surface, the adjacent liquid will evaporate so quickly that it is separated from the surface by a cushion of its own vapor. This state of affairs is referred to either as film boiling or the Leidenfrost phenomenon.

Film boiling falls into two general categories:

(1) Bulk or submerged film boiling is characterized by a continuous amount of liquid encompassing

the surface being cooled (a specimen placed in a liquid nitrogen bath).

(2) Leidenfrost film boiling is characterized by a discrete amount of liquid adjacent to the surface being cooled (drops and small puddles of liquid nitrogen on a surface).

As the surface cools, there comes a temperature below which stable film boiling terminates and transition boiling begins (at this point there is an enormous change in the heat transfer rates which significantly alters the freezing rates of tissue). This "minimum" surface temperature is referred to as the Leidenfrost or the departure-from-film-boiling temperature[1] for the liquid-surface combination. A certain amount of ambiguity exists in the experimental meaning of this point because it depends on many factors, chiefly the liquid itself and the thermal-mechanical properties of the surface. Consequently different experimenters measure different values depending on the surface conditions.

In this paper an attempt is made to determine the departure-from-film-boiling temperature (T_{DFB}) for pigskin cooled using liquid nitrogen. The difficulties encountered herein and variation in the determined values of this point classify the experiment as highly qualitative and much more work needs to be done.

Identification of the various temperature-heat flux combinations for a variety of specimens is a necessary step toward organ preservation. The cooling (and heating) rates of the most vulnerable tissue and cellular structures must be identified in order to maintain salt concentrations far below the lethal level (200 g/liter at -21.16° C).

EXPERIMENTAL DIFFICULTIES

The skin can be thought of as a stratified surface consisting of superficial epidermis and the dermis. While the thermal properties (eg. conductivity, specific heat, density, or thermal diffusivity) of some tissues have been determined (8 and 9) the variations of these properties with temperature down to 77 K and lower, are as yet unknown. For layered tissue such as skin, the thermal properties of each layer are needed[2].

One of the major thermal property problems is associated with the tissue structural changes such as the cream-color which appears on the epidermis as the temperature drops below the freezing point. High subcoolings (to -40° C) have been noted but once nucleation is initiated, the ice front travels at a temperature of -.56° C.[3] Interstitial freezing compared to complete freezing further complicates the determination of properties.

The effects of surface conditioning on the heat transfer rates even for metallic surfaces are not as yet, well understood. It is known that surface roughness, thin coatings such as grease and teflon , low diffusivity materials such as glass, and surface additives such as santocell (a fine powder) promote the destruction of the thin vapor film and affect departure out of stable film boiling into transition boiling at a much higher temperature. The effect of these agents on tissue is unknown, although one would anticipate similar results; i.e. roughness, grease, and hair follicles should promote vapor film destruction.

The topology of the surface being cooled also has a large effect on the cooling rates. For example fingerprints can be of the same order of magnitude as the vapor film thickness (2×10^{-4} cm). Wavy, conical, and vertical surfaces cause a transition from laminar to turbulent flow within the vapor layer. An increase in heat transfer rate and erratic vapor flow accompanies this transition.

The heterogeneity of the biological system is complex and not only complicates the determination of properties but alters the heat transfer rates. In-vitro and in-vivo rates differ chiefly because of blood flow, and need further clarification in determining the temperature for transition out of stable film boiling.

These factors make the classification of heat transfer regimes for tissue quite difficult to assess and apply to biological systems and cryosurgery.

EXPERIMENTAL EQUIPMENT

A thermocouple probe was constructed to be placed in the outer part of the epidermis. The probe was fabricated from 0.25 mm diameter swaged tubing and fit through a 24 gage hypodermic needle. The probe was positioned using the hypodermic needle which was then removed leaving the thermocouple embedded in the epidermis. The probe is illustrated in Figure 2. The chromel-alumel thermocouple was an open-ball type made from 0.038 mm diameter wire and sharpened to a point for further insertion. It is estimated that the thermocouple was within one diameter of the surface, and sensed a region less than one mm^3. Although the response of the probe to temperature variations was excellent, the "toughness" of the epidermis made insertion difficult.

The remainder of the equipment consisted of a strip chart recorder, spoon ladle for the application of liquid nitrogen to the surface, ice reference bath for the thermocouple probe and a piece of pigskin. The 12x20 cm piece of pigskin was taken from the hind leg and was "processed"; i.e., had gone through the boiling vat to remove the hair follicles etc., butchered, and cooled. The pigskin had no lesions, was white, and appeared very uniform.

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[1] For most surfaces the terms can be used interchangeably. Herein, departure-from-film-boiling (DFB) will be used.

[2] The epidermis alone consists of four distinct layers: stratums corneum, lucidum, granulosum, and germinativum.

[3] Dr. A. P. Rinfret: Private communication

RESULTS AND DISCUSSION

The needle was inserted just under the surface of the epidermis and the thermocouple pushed forward. The hypodermic needle was retracted and the thermocouple probe remained. One-half teaspoon (more or less) of liquid nitrogen was then ladled onto the surface above the thermocouple.

As the epidermis cools, a structural change occurs near 0^{o} C and the surface appears cream-colored. The cream-color characteristically occurs upon cooling below 0^{o} C and disappears upon heating back to 0^{o} C. Soon after the appearance of the cream-color the system changes from film boiling to transition boiling and then to nucleate boiling. Thus it appears that the cream-colored material promotes a very early transition; i.e., a very high departure-from-film-boiling temperature, and also signals the onset of a regime of very high heat transfer rates.

The hypodermic-needle "bores" a sizable hole in the skin and acts as a "foreign body" heat sink. Tests indicated that transition boiling initiated around the surface where the needle had been inserted and was greatly influenced by the length of retraction of the hypodermic needle. Consequently the probe is a first order disturbance of the system which is not damped out. In subsequent tests the procedure of insertion was modified. The hypodermic needle was inserted through the epidermis and run laterally for 1.2 to 1.5 cm through the dermis; the probe was then pushed up into the epidermis just under the surface and the hypodermic needle entirely retracted. The probe proved to be of inadequate rigidity and the removal of the hypodermic needle was tedious. Even with these precautions, the bore created by the needle still influenced the transition although the effect was greatly reduced. It is very difficult to measure the temperatures of the various strata of the epidermis without disturbing them (some sort of exclusion principle seems applicable).

A single value of temperature for the transition out of stable film boiling has not been obtained; however a most likely range of values was determined. The results are itemized in Table I.

RUN	T_L °K	PROBE PLACEMENT AND COMMENTS
1	272	Just under epidermis, hypo-needle retracted 1 mm, total insertion, 1 cm.
2	266	Parallel insertion to 1.5 cm and probe extended to epidermis--hypo retracted 1 cm.
3	243	As in 2, but hypo needle entirely retracted
4	261	As in 3. Large LN_2 puddle. Departure from film boiling initiated close to probe.
5	266	As in 3. Small LN_2 puddle. Color change to creamy followed by departure from film boiling
6	287	Surface Lesion (Probe installed as in 3)
7	242	Inserted probe through 4 cm of fat and dermis. Probe seated in epidermis with hypodermic needle removed.
8	270	As in 7. Surface was of irregular topology. Large puddle of LN_2. Probably areas of turbulent flow.
9	~287	Thermocouple probe "stuck" into the epidermis approx. 1 mm at an angle of .08 radians.
10	-	As in 7, plastic spoon ladle pressed against the skin.
11-13	-	Tests on authors middle finger, left hand
14	268	As in 7, irregular surface but of small "hills"
15	273	As in 7, shallow conical pit formed, probe at the bottom--surface punctured and smoothed.

Table I Transition from Stable Film Boiling Temperatures and Probe Placements for Pigskin

It should be noted that when the surface is flat, and disturbed very little by the probe, the departure-from-film-boiling temperature (T_{DFB}) is approximately 243 K. When the surface is nearly flat but there is some disturbance due to the probe, T_{DFB} = 260 to 275 K and for an irregular surface with little probe disturbance $T_{DFB} \approx 270$ K. Thus for practical applications it would seem that the departure-from-film-boiling temperature for pigskin is approximately 270 K. These values are much higher than the $T_{DFB} \approx 100$ K for a copper sphere immersed in LN_2, Fig. 1. The thermal diffusivity of tissue is about 1/45 that of copper, and tissue is therefore easily quenched. The epidermis is heterogeneous and of irregular surface roughness; however if one assumes it to be homogeneous and regular, a value of Leidenfrost temperature (T_L) can be estimated from reference 10. Within these assumptions $(T_L)_{est.} \approx (T_{DFB})_{est.} \leq 200$ K which is much greater than that of copper but less than that found herein for skin.

The cooling rates and the film boiling times for the runs of Table I are given in Table II and the profiles for run 3 are presented in Figure 3.

RUN	$\tau_{F.B.}$	$\left(\frac{dT}{d\tau}\right)_{F.B.}$	T_{DFB}	$\left(\frac{dT}{d\tau}\right)_{NB}$	
1	2.65	-6.95	272	-68.6	
2	8.1	-3.33	266	-37.2	
3	19.0	-1.7	243	-65.9	
4	7.85	-3.9	261	-53.6	Between 6.4 to 6.9 sec $dT/d\tau \approx 0$
5	11.6	- .83	266	-21.4	
6	~0	--	287	-82.7	
7	30.66	-1.94 - .794 - .715	242	-47.0	$26 < t < 30$ $7 < t < 14$
8	9.92	- .89	270	-64.0	
9			~287		T.C. probe stuck into the surface
10	~60	- .61	-	-	Plastic spoon pressed on surface
14	4.43	-5.84	268	-30.8	
15	7.84	-1.95	273	-137.0	

* 11, 12, 13 Finger cooling Data
DFB Departure from Film Boiling
F.B. Film Boiling
NB Nucleate Boiling
T Temperature, K
τ time, second

Table II Film Boiling Period, Departure from Film Boiling Temperature and Cooling Rates for Pigskin

The cooling rates vary considerably depending on the heat transfer regime (c.f. figs. 1 and 3); however note that they are dependent on the amount of liquid nitrogen on the skin, c.f. runs 4 and 5, thermocouple placement, c.f. runs 1, 2, 3 and 9, and surface topology, c.f. 14 and 15. Further tests isolating each of these parameters and associated cooling rates need to be performed.

One of the most pronounced effects on the departure-from-film-boiling temperature was the lesion. A simple scratch of the epidermis caused an almost immediate change from film to nucleate boiling. The estimated departure-from-film-boiling temperature was 287 K[4] with a cooling rate of -83 K/sec, see run 9, Tables I and II. The practical application of this fact cannot be overlooked. The surgeon can choose the areas of rapid cooling by

[4] The initial temperature was 287 K; higher film boiling transition temperatures would be anticipated for higher initial temperature levels.

tracing through them with the scalpel, or where lesions endanger the patient, very small particles such as carbon, metallic dust, etc. can be substituted for the scalpel trace. It also serves to caution one that cooling a surface with lesions or one which is "dirty" is completely different; the rates of cooling are high and the transition from film boiling is abrupt.

To obtain an idea of how the in-vivo system responded to liquid nitrogen, the author undertook some tests on his middle finger, left hand. The results of these tests are given in Table III.

RUN	τ_{min}	τ_{max}	$\tau_{most\ probable}$		Remarks
11	2.3	3.17	2.8		No cream color appears. Orientation: finger is nearly horizontal and only the lower part of the finger tip associated with the last of the metacarpol bones was in contact with liquid nitrogen. Alcohol wipe.
12	3.18	3.48	3.2		Orientation as in 11. Small cream patches occur at random on the surface, quenched in water, then dried.
13	4.	4.25	4.		Orientation as in 12. Cream color spread over most of the surface
15	1.38	1.18	1.28		Thorough cleansing with 70% alcohol. Orientation as in 11. Movie
16	1.98	1.88	1.9		Repeat of 15 for a longer time period. Movie
17	2.52	2.44	2.45		Repeat of 16 Movie
18	2.81	2.31	2.4	⊕	Repeat of 17 Movie
19	3.78	3.66	3.7		Same as 15. Cream color appears but is weak. Movie
20	5.72	4.72	5.		Cream color spreads over the surface, same as 13. Movie
21	2.6	2.1	2.2		Lesion effects Movie
22	1.98	1.71	1.8		Lesion effects Movie
23			.82		Vertical finger plunge--the fingernail becomes cold very quickly and serves as a fin and a path for LN_2 to wet the skin.
24			1.02		Vertical finger plunge--"hangnail" quite close to the nail begins to turn cream colored and the patch beings to spread.

⊕ dirt particle on the skin

Table III Some in-vivo Effects of Liquid Nitrogen on Tissue

Again the most significant effect was that of a small lesion and "dirt" particle. Transition boiling began at these points and a pronounced cream-colored region formed about these centers. The cooling sensation with the "dirt" particle was less than with the lesion as would be expected. The lesion gave the sense of sharp burning pain which was highly localized and disappeared as the surface warmed.

As noted in Table III, cream colored spots appeared after about 3 seconds and spread over the entire lower part of the fingertip in 4 to 5 seconds. (The author then dunked the finger in water). After the 5-second test a white spot encompassed by a reddened ring was noted. The spot was quite sensitive to heat for several hours after the experiment. Such spots were noticeable for several days.

To illustrate the effects of orientation, the author placed the fingers in a liquid nitrogen bath in the vertical direction, see Table III. The fingernail and "hangnails" cool very rapidly and cream coloring appears in about 1 second.

It seems quite reasonable to expect laminar heat transfer when the finger is placed horizontally in a liquid nitrogen bath and laminar to turbulent heat transfer when the finger is vertical. It also seems reasonable that dropping liquid nitrogen on a "hairy" surface would give the same type of reaction as a lesioned surface.

A motion picture supplement has been made to illustrate the effects listed in Table III for those runs so marked.

From the effects noted in the movie and those on the pigskin it seems that in-vitro and in-vivo results differ primarily in the blood supplied as an energy source.

For a cryogenic system, molding and contouring for preservation and destruction is a field in itself. Such things as plastics and flexible wire contour devices can be used to control cooling levels and rates. As an example, in run 10 the plastic ladling spoon was pressed against the pigskin. While boiling took place within the spoon reservoir, the cooling rate was uniform at -0.61 K/sec for over 50 seconds. (The liquid nitrogen evaporated). As a consequence, variations in container thermal properties can be used to control cooling rates and combinations of these with lesions or surface particles should lead the user to a variety of cooling devices.

CONCLUSIONS

In cooling experiments using in-vitro pigskin and liquid nitrogen as the coolant, the following results were noted:

1. A cream-colored surface appeared at a temperature less than 273 K; furthermore as the surface was warmed the cream color disappeared at about 273 K.
2. A special probe was fabricated to measure surface temperatures; however it was found that the probe introduced a first order disturbance unless inserted from the dermis into the epidermis.
3. The temperature of transition from stable film boiling (T_{DFB}) for a flat surface which is not greatly disturbed by the probe was approximately 243 K. For disturbed surfaces of varying topology the transition temperature ranged from 260 to 275 K.
4. For lesions, the transition out of film boiling was immediate and T_{DFB} was approximately the initial temperature, in this case 287 K.
5. The quantity of nitrogen used and orientation (surface topology) had a pronounced effect on cooling rate.
6. In-vivo experiments with a finger seemed to be in agreement with the in-vitro results, except that the surface temperatures were not recorded and transitions spread very slowly due to the blood heat source. A motion picture of these effects was made.

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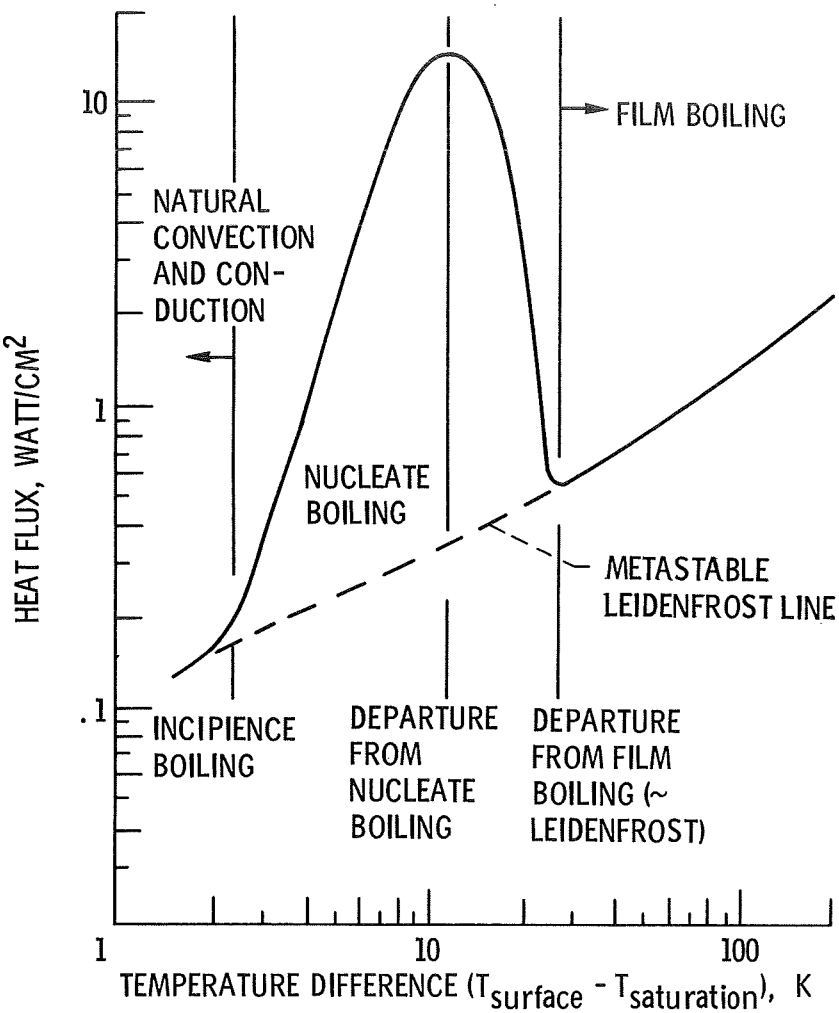

Figure 1. - The boiling curve for nitrogen illustrating the various heat transfer regimes (ref. 11).

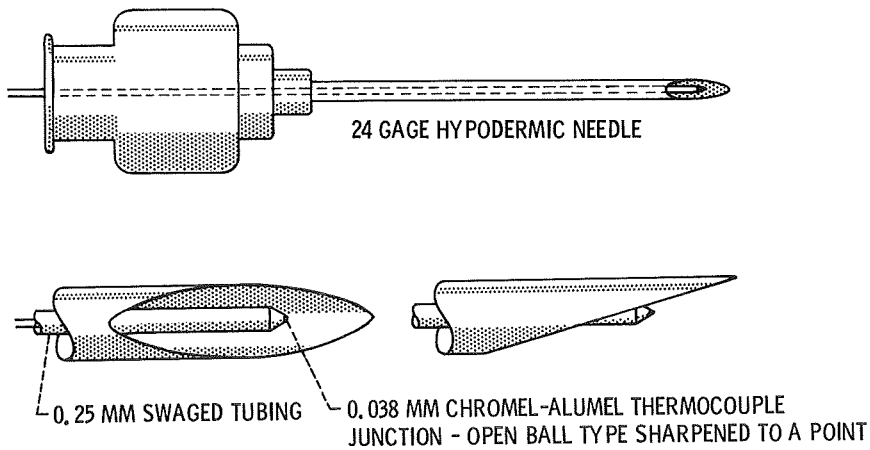

Figure 2. - Surface temperature probe.

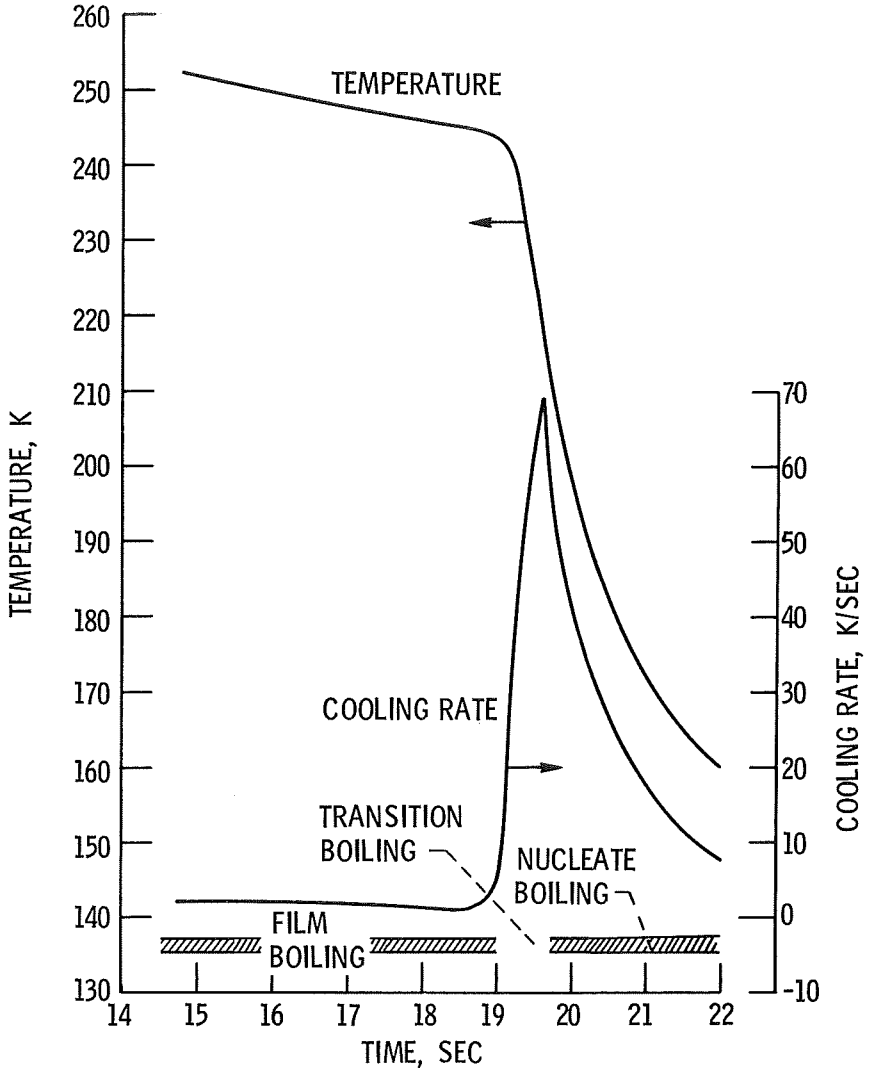

Figure 3. - Temperature and cooling rate profiles for liquid nitrogen on pigskin - run number 3 of table I.