TRANSITION FROM HALL-PETCH HARDENING TO SUPERPLASTIC BEHAVIOUR

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ABSTRACT

Transition from Hall-Petch region to superplastic region has been established in the case of two commercially used superplastic alloys *viz.* Zn-Al eutectoid and Al-6Cu-0.5 Zr alloys. Tensile tests at various temperatures and for different grain sizes have been utilized for the experiment.

INTRODUCTION

THE field of application of superplastic materials is currently becoming prominent. Investigations are in progress to develop new alloys and to commercially use the already developed ones. Most of the alloy systems exhibit two opposing characteristics in the ambient and elevated temperatures. These materials are strong for practical purposes at lower temperatures and weak enough for forming operations at higher temperatures. Hence it is of interest to know the region of transition for each alloy system so that it may be used more efficiently.

In the present age requiring energy conservation, energy saving operations assume special importance. Superplastic forming can be assumed to be one such operation. It is in this context that the need for the characterization of different superplastic materials, in terms of their properties and behaviour under different conditions, arises.

The commercial use of a superplastic material lies in its ability to behave like a conventional metal or alloy within a particular temperature range characteristic of the metal or alloy. In this range of temperature, the concerned metal or alloy generally satisfies the Hall-Petch relationship[1,2] *viz.*

$$\sigma_y = \sigma_i + K d^{-1/2} \quad (1)$$

where σ_y is the tensile stress and d is the grain diameter. σ_i and K are constants at a specified temperature. In hcp metals, σ_i and K are observed to be strain-rate-dependent also, whereas in fcc and bcc metals only σ_i is strain-rate dependent.

An empirical relation[3,4] connecting flow stress, grain size, strain rate and temperature holds good in the superplastic temperature region. This is given as

$$\varepsilon^\circ = A\frac{\sigma^n}{d^a}\exp(-Q/kT). \quad (2)$$

From (2), at constant strain rate (ε°) and temperature (T), it can be shown that

$$\sigma_{(\varepsilon^\circ, T)} = Bd^b, \quad (3)$$

assuming that the activation energy (Q) for the mechanism remains the same. Here B and b are constants. In the case of Zn-Al eutectoid, b is found[5] to be equal to 1.

If σ_1 and σ_2 are the stresses for corresponding grainsizes d_1 and d_2 respectively at constant strain rate and temperature, then from (3), it can be obtained that

$$b = \frac{\ln(\sigma_{1^\circ}/\sigma_{2^\circ})}{\ln(d_1/d_2)} \quad (4)$$

In this paper, the attempt to find out the transition temperature between Hall-Petch region and the region where the material shows superplastic behaviour is illustrated. A knowledge of such a temperature range is necessary to set the safe limit for the use of a superplastically-formed component and to fix the temperature for the forming operation.

EXPERIMENTAL

Zn-Al Eutectoid Alloy. This alloy was cast in the normal way. Excess zinc had been taken for melting to compensate for the zinc loss during melting. The cast bars were homogenized at 620 K for 8 hr. Samples were taken from these bars to analyze for Al content and for impurities.

The bars were then hot-rolled at 520 K. The rolling reduction was about 90% to achieve a thickness of about 1.25 mm. Tensile test specimens were made from the rolled sheets. The gauge length of these specimens was 13 mm and the width along the gauge length was 3 mm.

To achieve fine grains, the specimens were subjected to heat treatment, consisting of solutionizing at 620 K for 20 hr and quenching in ice water. After quenching, the total number of specimens was divided into four equal lots. Each lot was given aging treatments at

520 K for different periods of time *viz* 1/2, 1, 2 and 4 hr. This was done to get different grain sizes.

The heat-treated specimens were subjected to tensile testing on a modified Hounsfield tensometer using oil bath for high temperature tests. Tests were carried out at 301, 323, 373, 423, 473, 493 and 523 K.

The ultimate loads were determined from the graph obtained for each test. From a knowledge of the load and dimensions of the gauge length, the ultimate stresses were calculated. The cross head (CH) speed was maintained constant by keeping a constant gear setting for the motor. This setting corresponded to the stress rate specified for superplastic deformation (5.9×10^{-4} sec^{-1}).

Earlier work[6] reported that there was no significant change in grain size after superplastic deformation, the grain sizes were determined by linear intercept method.

Al-Cu-0.5 Zr Alloy: This alloy was obtained in sheet form from the Department of Metallurgical Engineering, I.I.T., Madras. The sheet was further reduced in thickness by about 12% by hot rolling at 573 K. Specimen preparation was done in a similar way as done for Zn-Al alloy.

Heat treatment consisted of maintaining the temperature at 670 K for 1, 3, 6 and 12 hr differently for each lot. They were then quenched in ice water and a stress relieving treatment was given at 470–520 K for 1 hr.

Tension tests were conducted at 301, 373, 473, 573, 623 and 673 K. Temperatures above 573 K were obtained using air as atmosphere. Grain size measurements were carried out on polished and etched samples. Etching was done using Keller's reagent.

RESULTS AND DISCUSSION

The Zn-Al alloy had an Al content of 21.4% by wt., since near-eutectoid alloy also has been reported to exhibit superplasticity[7], no further attempt was made to make the present alloy achieve the exact eutectoid composition.

The spectrographic analysis for impurities gave the following results:

Element	Cu	Mg	Fe	Si
Weight %	0.6	0.02	0.1	0.02
Pb	Cd	Sn	Th	Others
0.004	0.002	0.003	0.001	0.006

The amount of impurities is comparable with that given for a commercial superplastic Zn-Al eutectoid alloy[8].

The grain sizes obtained depended on the ageing time given as the temperature was fixed. The longer the time, the larger the size of the grains.

The results obtained from tensile tests are plotted as stress-temperature diagram (figures 1, 2). In both the figures two temperature regions can be identified. In

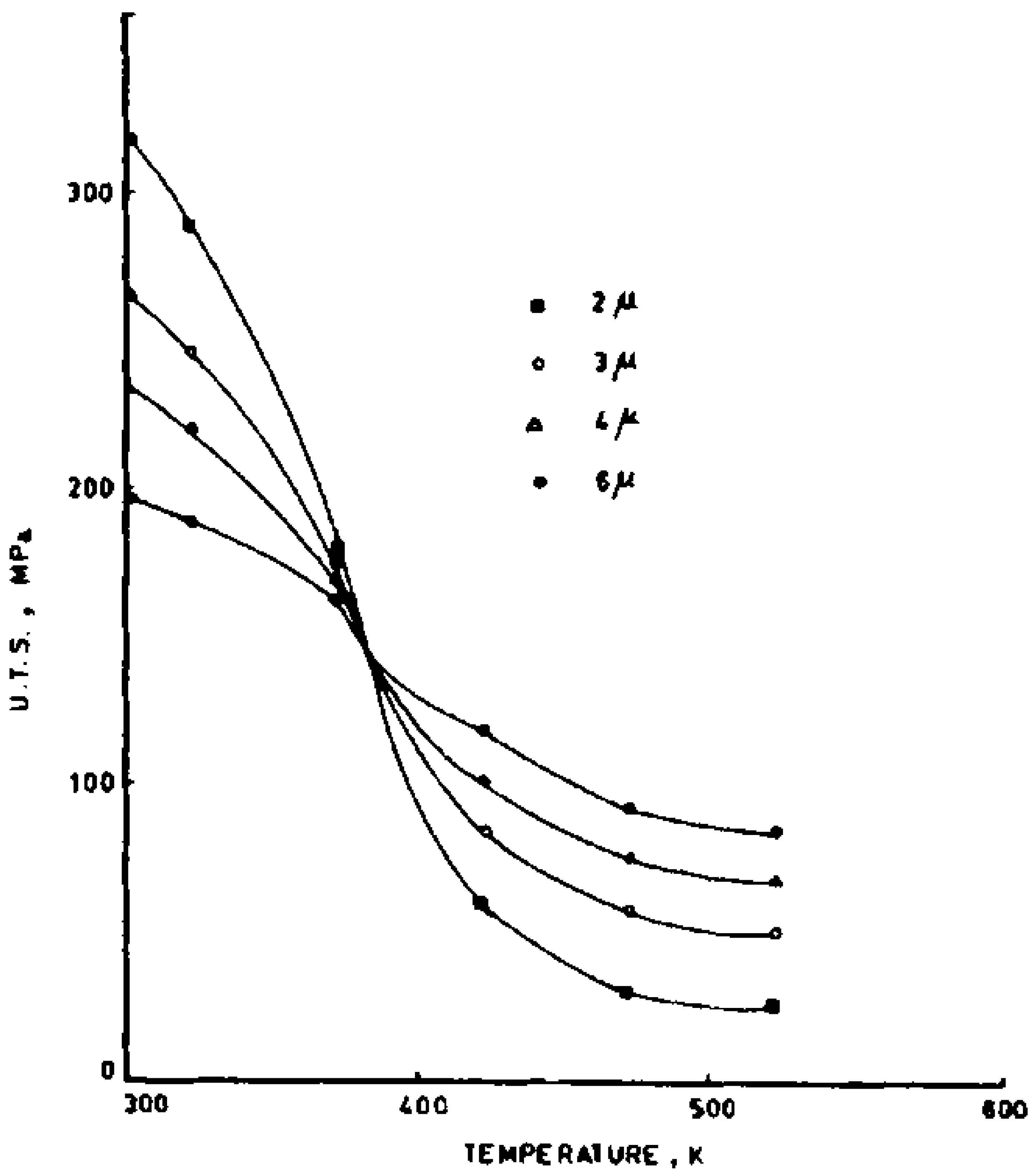

Figure 1. U.T.S. *vs* temperature diagram for Zn-Al system.

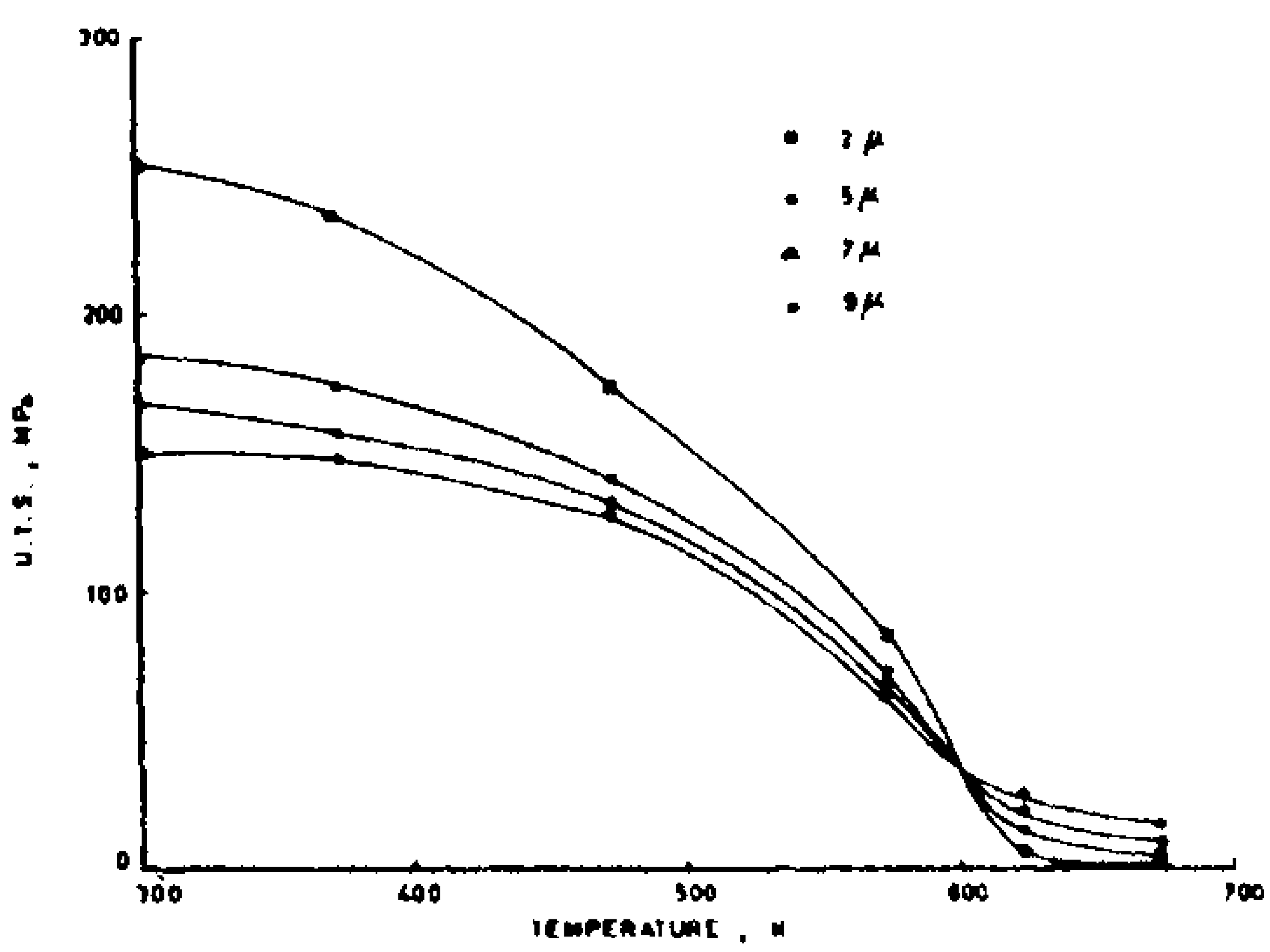

Figure 2. U.T.S. *vs* temperature diagram for Al-6Cu-0.5 Zr system.

the lower temperature region, the following properties can be ascertained.

(i) The stress decreased with temperature, for a specific gram size
(ii) Stress decreased with increase in grain size at a fixed temperature

The second property is consistent with Hall-Petch law (equation (1)). Hence this region can be termed as the Hall-Petch region. The results have been verified using (1) (table 1).

In the higher temperature region, the following properties can be identified:

(i) The stress decreased with an increase in temperature for a specific grain size.
(ii) The stress increased with an increase in grain size, temperature remaining constant.

The first property is common for both the regions. The second is associated with superplasticity, hence this region may be termed as superplastic region. High temperature creep differs from superplasticity in that the strain rate for creep is extremely low. The results have been verified using (2) (table 2).

There is a narrow temperature range (almost a point) between the two regions, which may be termed as the transition region.

Table 1 *Verification of the results obtained in the Hall-Petch region*

I. Zn-Al Alloy

Temp K	σ_1 (MPa)	σ_2 (MPa)	d_1 (μ)	d_2	$\sigma_i = \frac{\sigma_1\sqrt{d_1}-\sigma_2\sqrt{d_2}}{\sqrt{d_1}-\sqrt{d_2}}$ (MPa)
301	319	266	2	3	30
	266	234	3	4	27
	234	196	4	6	27
323	290	246	2	3	50
	246	220	3	4	52
	220	188	4	6	46
373	180	172	2	3	136
	172	168	3	4	146
	168	162	4	6	135

II. Al-6Cu-0.5 Zr

301	254	186	2	5	69
	186	164	5	7	44
	164	150	7	9	45
373	236	174	2	5	67
	174	158	5	7	71
	158	148	7	9	73
473	174	140	2	5	81
	140	132	5	7	88
	132	128	7	9	98
573	84	70	2	5	46
	70	66	5	7	44
	66	62	7	9	32

Table 2 *Verification of the results obtained in the superplastic region*

I Zn-Al Alloy

Temperature K	Ultimate stress, σ (MPa)	Grain size, d (μ)	$B = \sigma/d$
423	60	2	30
	84	3	28
	102	4	26
	118	6	20
473	30	2	15
	58	3	19
	76	4	19
	92	6	15
523	26	2	13
	51	3	17
	68	4	17
	84	6	14

II Al-6Cu-0.5 Zr

Temp. K	σ_1	σ_2	d_1	d_2	$b = \frac{\ln(\sigma_1/\sigma_2)}{\ln(d_1/d_2)}$
623	6	14	2	5	0.92
	14	20	5	7	1.06
	20	26	7	9	1.04
673	2	6	2	5	1.20
	6	10	5	7	1.52
	10	16	7	9	1.87

SUMMARY AND CONCLUSION

Transition from Hall-Petch hardening to superplastic behaviour has been established by means of tension tests at various temperatures and for different grain sizes. The transition occurs over a narrow temperature region almost at a point. This point may be termed as the temperature of inversion.

20 July 1985; Revised 25 November 1985

1. Hall, E. O., *Proc. Phys. Soc.*, Ser. B., 1981, **64**, 747.
2. Petch, N. J., *J. Iron Steel Inst.*, 1953, **174**, 25.

3. Holt, D. L. and Backohen, W. A., *Trans. ASM.*, 1966, **59**, 755.
4. Packer, C. M. and Sherby, O. D., *Trans ASM.*, 1967, **60**, 21.
5. Ball, A. and Hutchison, M. M., *Metal Sci. J.*, 1969, **3**, 1.
6. Nuttall, K., *J. Inst. Met.*, 1972, **100**, 114.
7. Holt, D. L., *Trans. Met. Soc., AIME*, 1968, **242**, 7.
8. Yadava, R. K., *A unified approach to superplastic deformation*, Ph.D. thesis, BHU, 1982.

ANNOUNCEMENTS

INDIAN SOCIETY FOR THERMAL SPRAYING

At a meeting organized by Indian Lead Zinc Information Centre at Centaur Hotel, Bombay on 17 December 1985, the 'Indian Society for Thermal Spraying' was formally inaugurated by Mr. A. C. Wadhawan, Chairman-Managing Director, Hindusthan Zinc Limited, Udaipur.

Membership of the Society is open to thermal sprayers, users industries, equipment manufacturers, consumable manufacturers, research laboratories etc. Those interested in joining as members should write to Indian Lead Zinc Information Centre, B-6/7, Shopping Centre, Safdarjung Enclave, New Delhi 110029.

INDIAN INSTITUTE OF SCIENCE ALUMNI ASSOCIATION— 'DISTINGUISHED ALUMNI AWARDS'

The 'Distinguished Alumni Awards' instituted by the Indian Institute of Science Alumni Association, Bangalore, are given annually to a distinguished alumnus selected jointly by the Institute Governing Council and the Alumni Association.

Prof. C. N. R. Rao, Director of the Indian Institute of Science and President of the Current Science Association, received the 1983 award. Prof. Satish Dhawan, Senior Advisor to the Department of Space, former Director of the Institute, received the award for 1984.

Sri T. R. Satish Chandran, Chief Secretary to Government of Karnataka, Bangalore and an old boy of the Indian Institute of Science read out the citations.

INDIAN SOCIETY OF DEVELOPMENTAL BIOLOGISTS

The Indian Society of Developmental Biologists has awarded to Professor Sivatosh Mookerjee FNA, School of Life Sciences, Jawaharlal Nehru University the SPS Award for Developmental Biology in recognition of his pioneering contributions to teaching and research in developmental biology, particularly in the areas of morphogenesis and differentiation in the chick, *Hydra*, sponges and protozoans. The award carries a cash prize of Rs.3,000/- and a citation.