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中国科学院金属研究所

INSTITUTE OF METALS RESEARCH, ACADEMIA SINICA

金属学与金属物理

metallography and metal physics

材料研究

materials research

新技术与新工艺

new technologies and processes

应用与推广

application(s) and development(s)

前 言

PREFACE

中国科学院金属研究所创建于1953年。位于我国重工业基地——沈阳，是我国主要的材料科学与工程研究机构之一。

三十年来，在科研工作中取得了显著成绩。在研制高温合金、难熔金属、特种合金钢、碳素材料等新型材料方面；在发展焊接、真空冶炼、表面喷涂等工艺方面；在研究金属晶体结构与缺陷、组织结构与性能、形变与断裂、氧化与腐蚀、钢中气体与夹杂物、金属内耗等方面做了大量工作，发展了有关金属合金的基础理论和实验技术，为生产建设提供了许多具有重要价值的成果。

金属所努力加强国际学术交流，与美国、英国、西德、日本、法国、意大利等国家的科学研究机构和学术团体进行了广泛的接触与交往，不断增进各国科学家之间的了解与友谊。

目前，金属所有研究员9人，副研究员43人，高级工程师13人，其中包括中国科学院技术科学部委员师昌绪、郭可信、庄育智等知名科学家。有科技人员600余人。还有一批研究生正在勤奋地攻读博士学位和硕士学位。

金属所拥有各种高性能的先进仪器和设备，诸如高分辨电子显微镜、场离子显微镜、电子探针、多通道声发射仪，电液伺服疲劳试验机，电子束炉、真空感应炉、激光热导仪、特种焊接装置、氮、氮液化设备等。图书馆藏有科技书籍七万四千册，订有中外科技期刊873种。

金属所创建人，长期担任金属所所长的李薰教授不幸于今年三月逝世，但他对金属所立下的功绩将永不磨灭。现任所长是师昌绪教授。

金属所全体职工正团结一致，努力把金属所建成一个具有先进水平的研究机构，为发展科学事业和振兴中华做出贡献。

The Institute of Metal Research, Academia Sinica was originally established as a centre for the study of Metallurgy in 1953 and grew rapidly in the past thirty years to become one of the largest research centers for materials science and engineering in China. It is now situated in Shenyang, an important industrial city in this country.

During the past thirty years, significant progress in the Institute has been made. Much has been done in the development of new materials such as superalloys, refractory metals, special alloy steels, and carbon materials, and in working out of new technologies such as welding, vacuum metallurgy, and surface spraying, and in the study of crystal structure and defects in metals, structure and properties, deformation and fracture, oxidation and corrosion, gases and inclusion in steels, and internal friction in metals. In addition, the basic theory for metals and alloys and new experimental techniques have been developed. As a result numerous significant contributions to industries and to national economy have been made.

In recent years efforts have been made to promote the international academic exchanges and to keep close contact with scientific research institution and learned societies in the United States, Britain, West Germany, Japan, France, Italy, Romania etc to deepen friendship and understanding among scientists of these countries.

The academic and technical staff in this institute are more than six hundred, including nine professors, forty-three associate professors and thirteen senior engineers. Among them are Prof. Shih Chang-xu Prof. Guo Kexin, and Prof. Zhuang Yuzhi, members of the Technological Science Division of Academia Sinica. Under their guidance many postgraduate students are industriously pursuing for degrees of Master or Doctorate in science.

The institute is well equipped with various kinds of advanced apparatuses and instruments such as high resolution electron microscope, field ion electron microscope, electron probe, multi-channel acoustic emission source location and analysis system, electro-servo hydraulic fatigue testing machine, electron-beam furnace, vacuum induction furnace, laser pulse thermal conductivity equipment, special welding equipment, nitrogen and helium liquefiers and so on.

There are 74,000 copies of scientific and technical books collected in our library with 873 kinds of journals and periodicals issued at home and abroad.

Prof. Lee Xun, who founded this Institute and was director for many years, died in March this year, but the contributions he had made to the Institute will be always remembered. Prof. Shih Changxu is now in charge of the Institute.

All the personal in our Institute will work enthusiastically to raise the academic standard of the Metal Research Institute and to make further contributions for the cause of developing science and technology and for the modernization of our country.

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1. 金属学与金属物理

METALLOGRAPHY AND METAL PHYSICS

1-1 晶体结构和晶体缺陷

CRYSTAL STRUCTURE AND CRYSTAL DEFECTS

由三方面组成：1. 固体原子像的观察。利用 JEM-200CX 高分辨电镜对金属、金属氧化物、无机化合物、矿物及催化剂等固体材料的原子分布进行直接观察以研究其结构及缺陷。并配有场离子显微镜——原子探针设备。

2. 表面与界面的研究。应用表面科学测试手段研究固体表面及界面在各种条件下的结构与成份，以探索气体吸附、杂质偏析规律。配有俄歇谱仪。

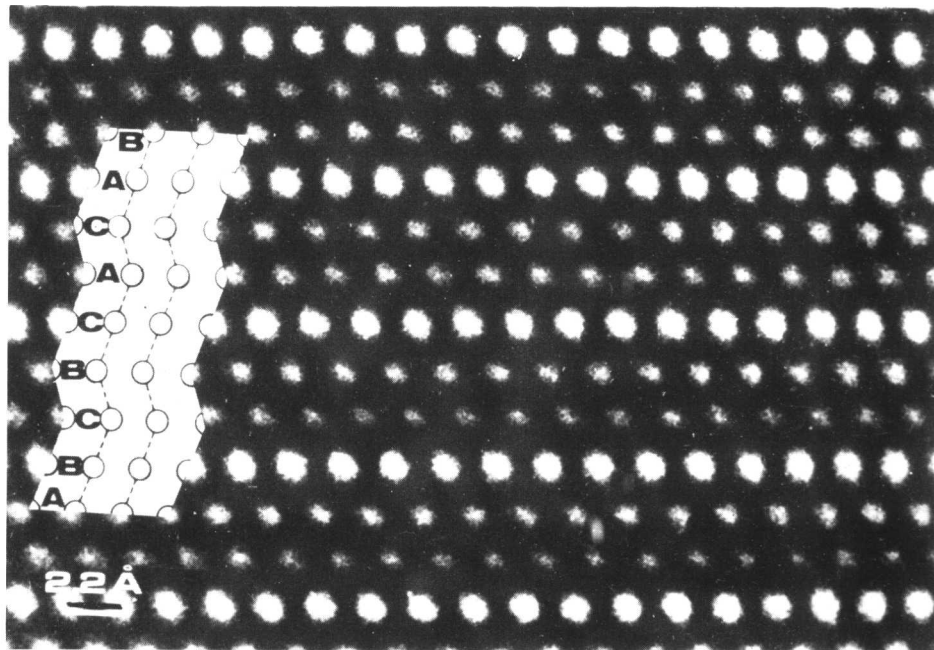
3. 非晶态晶化相的研究。研究非晶态，晶化前期，晶化亚稳相及晶化相的结构与缺陷。

The study of crystal structure and defects consists of:

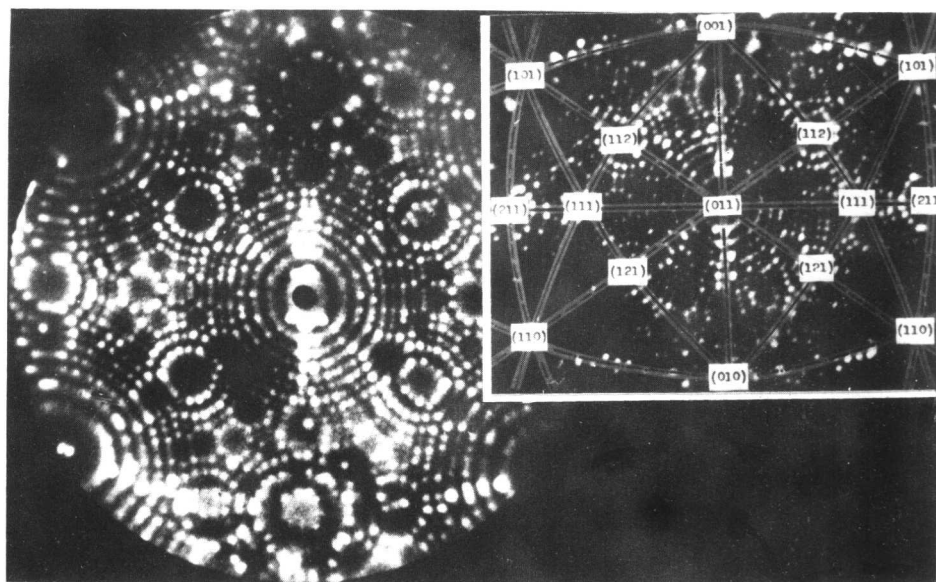
1. High resolution electron microscopy: Structural image at atomic resolution of solids such as alloys, oxides and other inorganic materials, catalysts, minerals found recently in China, etc. There is a high resolution electron microscope JEM 200CX.

2. Applied surface science: Surface and interface structure and composition under various conditions such as absorption of gases, intergranular embrittlement, vapour deposition, etc. There is an AES-LEED and an FIM-Atom Probe.

3. Structure of amorphous materials and their crystallization processes, especially the transition structures and metastable phases.



High resolution image of 9R long period structure in the Ni_3 (TiV) alloy along $[0i0]$ orientation



Field ion image of $[011]$ - oriented tungsten

1-2 缺陷与金属的力学性质

DEFECTS AND MECHANICAL PROPERTIES OF METALS

金属所在金属物理方面的研究有着悠久的历史，目前的研究领域主要有：

断裂物理，内耗与超声衰减，金属中的电子和声子，金属中缺陷的正电子湮没和穆斯堡尔谱研究。

近年来，恢复了基础研究，利用缺陷电子态的正电子湮没效应研究了裂纹顶端范性区的多普勒展宽变化；改进 BCS 位错模型，计算了范性区位错密度分布，试制成一台真空自动倒扭摆装置；还继续进行了断裂韧性的测试和应用研究。

Research fields: Defects and mechanical properties of metals, fracture physics, internal friction and ultrasonic attenuation, electrons and phonons in metals, positron annihilation and Mössbauer effects of defects in metals.

Fundamental research, such as positron annihilation in the plastic zone at a crack tip and the calculation of distribution function of dislocations in the plastic zone using a modified BCS Model have been carried out. A computer controlled automatic torsion pendulum has been developed.

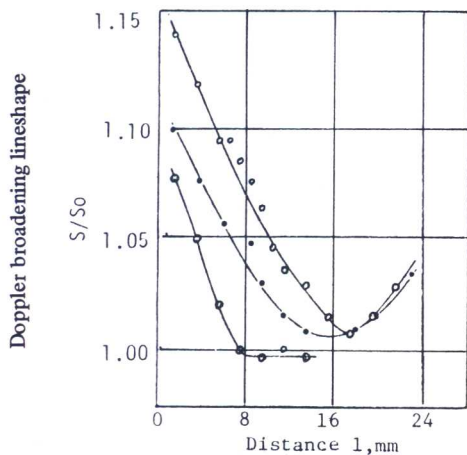


Fig. 1.2.1 Relative change of Doppler broadening lineshape of positron annihilation as a function of distance from crack tip in α -Ti.

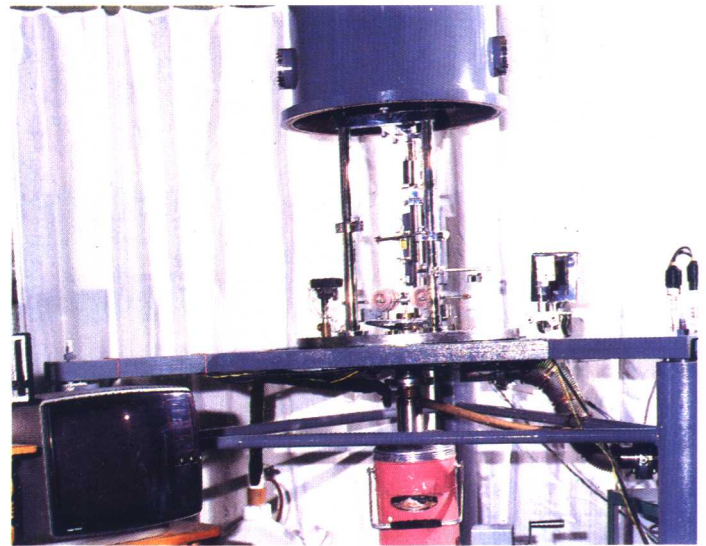


Fig. 1.2.3. Automatic torsion pendulum controlled by computer

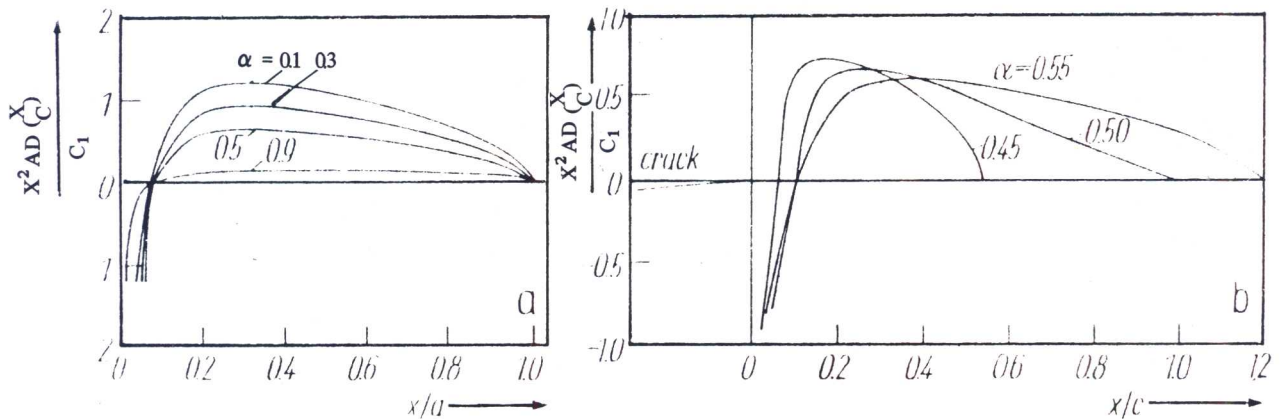
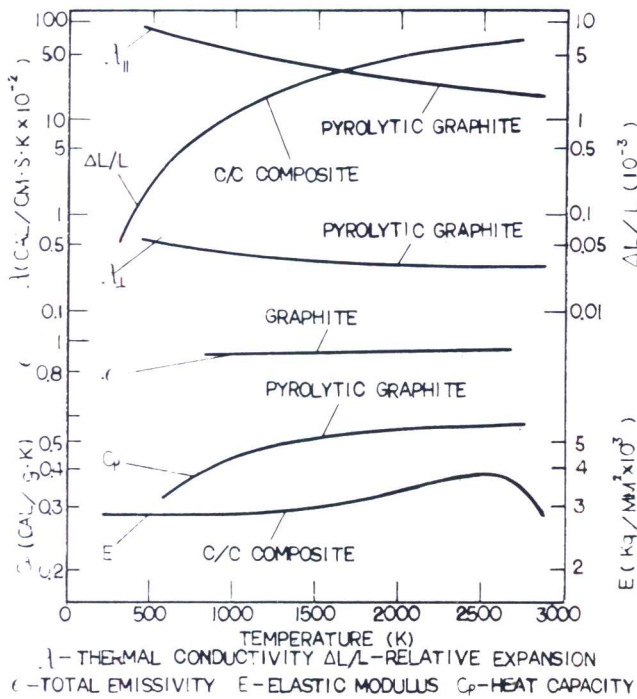


Fig. 1.2.2 Dislocation distribution in the plastic zone under small scale yielding, a) x/a unit, b) x/c as unit.

1-3 固体物理性能

PHYSICAL PROPERTIES OF SOLIDS



Variation of physical properties of solids with temperature

为准确测定金属、非金属、涂层和复合材料等的高低温热物理性能，发展了热导、膨胀、比热、弹性模量和辐射率等一系列测定装置，并在测试原理上有所创新。从实验上和理论上解决了材料在烧蚀过程中形状和结构变化时的热导测定问题。我所研制的远红外节能涂层已在辽宁和福建等地推广。目前正进行超导、夹层化合物的热电势率，以及晶格振动和热膨胀的微观动态过程物理本质的研究。

研究领域已从热物理性能逐步扩大到其它性能，包括测试方法，实际应用和基础理论三个方面的内容。

A series of equipments has been developed for measuring precisely the thermophysical properties, such as thermal conductivity, thermal expansion, specific heat, elastic modulus and radiative emissivity of metals, non-metals, coating and composite materials over a wide range of temperature. Some new principles for measuring are suggested. The measuring of thermal conductivity of materials with changing shape and structure during ablation has been solved both experimentally and theoretically. Far-infrared coatings developed in this Institute for energy saving have been manufactured and utilized successfully in practice in Liaoning and Fujian provinces. Recently, some studies on super-conductivity, thermoelectric power of intercalation compounds and the physical mechanism of properties such as lattice vibration and the microscopic dynamic process of thermal expansion are in progress. The field of study has been enlarged gradually from thermophysical properties to other properties and includes the studies on methods of measuring, practical applications and basic theories.



Laser pulse equipment for measuring thermal conductivity



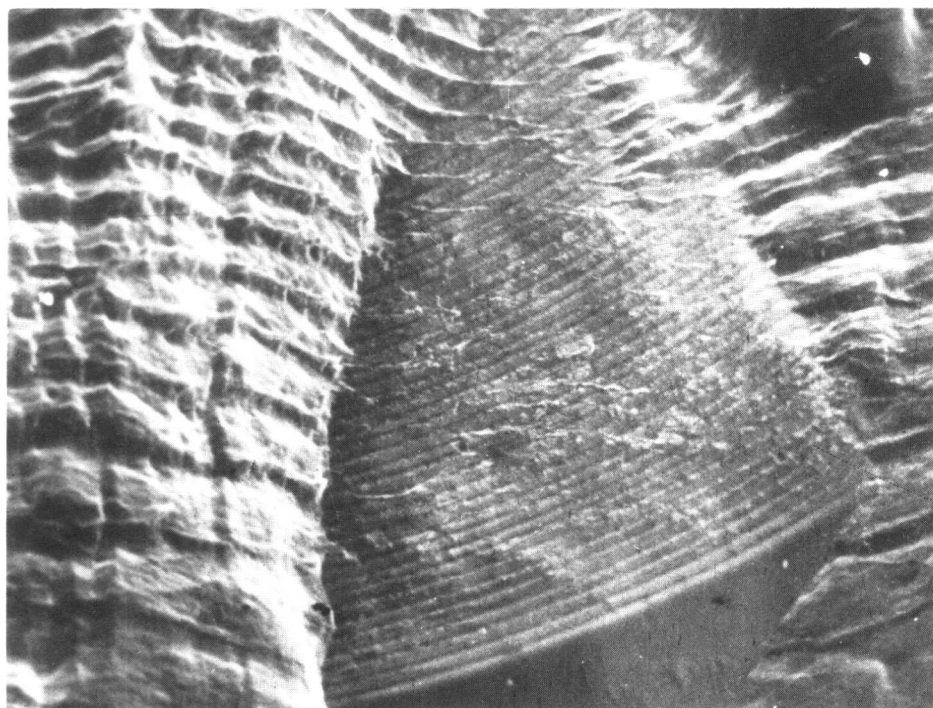
Auto-adiabatic equipment for measuring specific heat



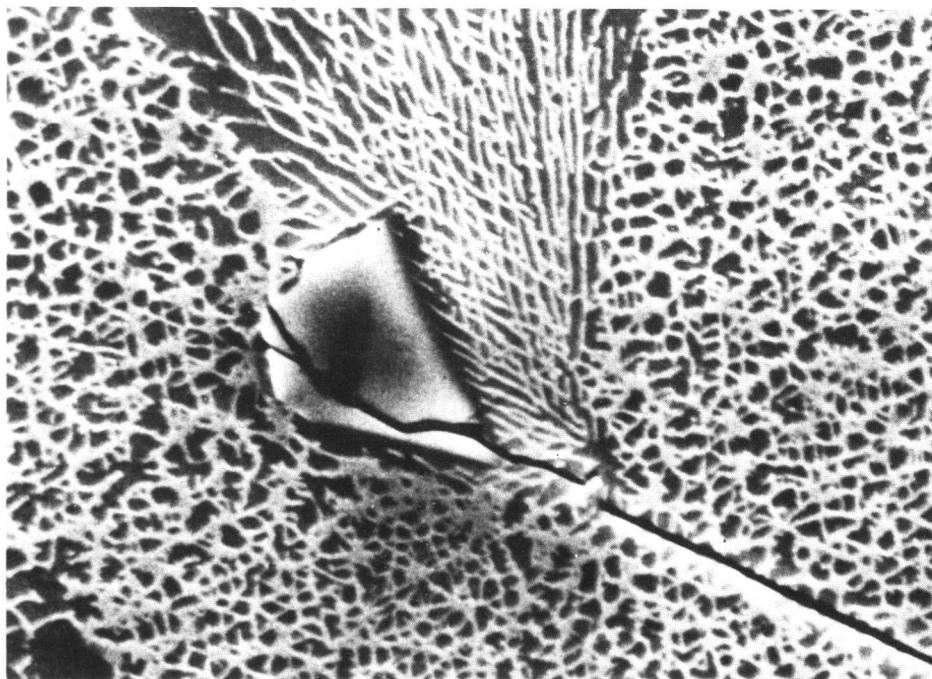
Auto-recording equipment for measuring thermal emissivity

1-4 疲劳断裂

FATIGUE FRACTURE



Research on mechanism of fatigue of pure Al. Fatigue crack growth rate shown on fracture surface.



Near-threshold fatigue propagation in Ni-base superalloy. Observation under SEM

从60年代初开始，金属研究所逐渐建立了从高频到低频，从简单载荷到随机载荷、从高温到低温，从空气介质到腐蚀介质的疲劳实验室，并以发掘材料性能潜力为目标，开展了疲劳物理原理，工程材料低周疲劳寿命预测，表面残余应力和表面强化工艺等项研究，取得了明显的经济效益。近年来工作集中于面心立方金属单晶及复相材料循环应力应变行为，疲劳裂纹的形成和初期扩展，冶金因素及环境介质对疲劳门槛值的影响，过载效应以及蠕变疲劳交互作用等方面。

The fatigue research laboratory was founded in early sixties and continued to grow carrying out alternating tests at different temperatures and frequencies in various environments as well as fatigue test with random loading. In order to make the most of the potential mechanical properties of materials, fatigue mechanism, low cycle fatigue behaviour of superalloys, evaluation of residual stresses and the effects of surface treatment on fatigue have been investigated in the laboratory and considerable economic benefits have been achieved.

Current work in the fatigue lab. is concentrated on the following programmes: cyclic stress-strain behaviour of FCC single crystals and dual phase materials; initiation and propagation of fatigue crack at early stage; effects of metallurgical and environmental factors on fatigue threshold; overload retardation and creep-fatigue interaction.

1-5 断口学与失效分析

FRACTOGRAPHY AND FAILURE ANALYSIS

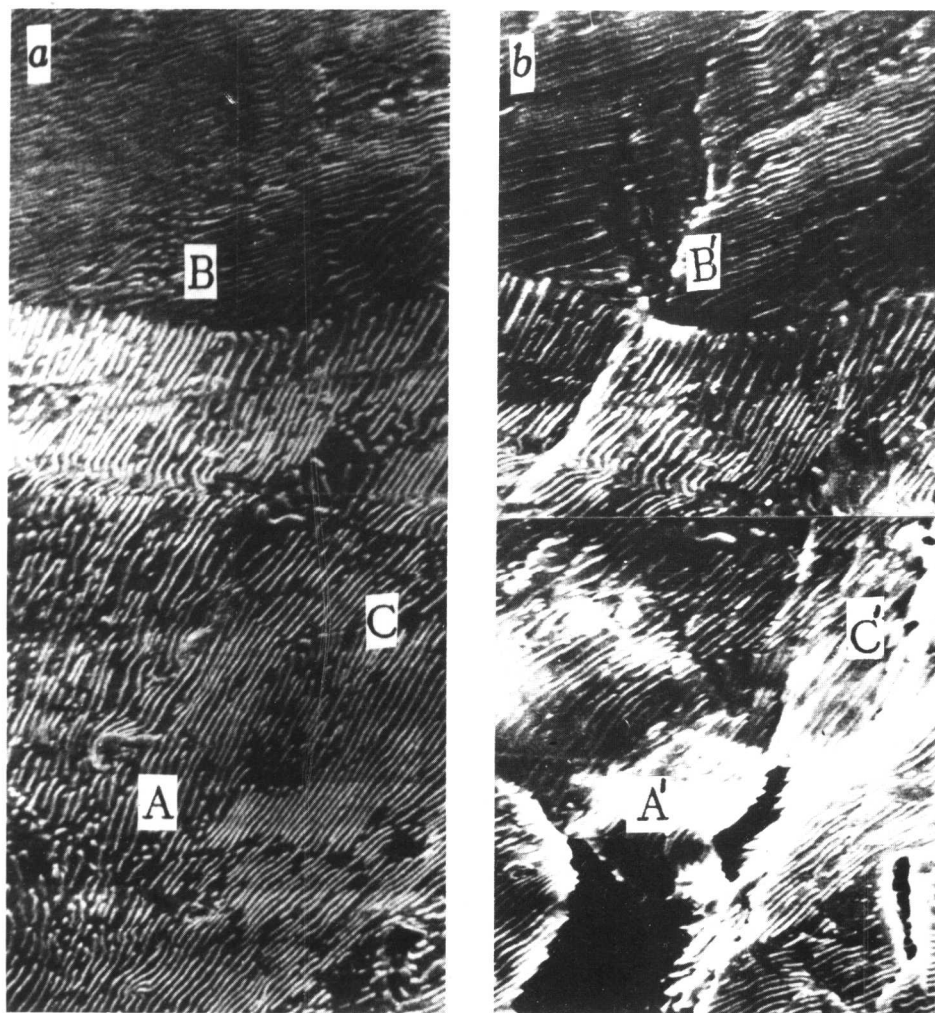


Fig. 1.5.1. shows the dynamic process of nucleation and propagation of crack and fracture in pearlite structure in which the crack initiated at pearlite colony interface (B) and growth fault (A, C). As applied stress increases, the main crack (at A') coalesces with the microcrack ahead and finally fracture forms.

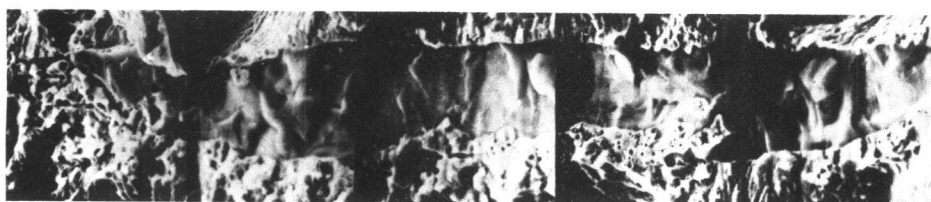


Fig. 1.5.2. shows a mechanism of flake formation in steels. The growth mechanism without a nucleation process and with a microvoid as a starting point of flake growth has been suggested after a study on dynamic process of flake formation. Through cooperation with the Anshan Iron Steel Company, a new appropriate technological process has been taken to remove flakes and good economic benefit has been achieved in production.

利用电子光学仪器、声发射、超声波等技术综合研究氢致损伤和断裂的微观过程，发展断口学，并用以指导冶金质量控制及工程断裂事故分析。十多年来断裂与失效分析研究为我国航空、石油化工、机械工业以及公安侦破案件等方面都做出了重要贡献。

Hydrogen damage and fracture micro-process were investigated by means of electron optical instruments, acoustic emission and ultrasonic techniques. Fractography was developed for controlling qualities of metallurgical products, as well as for failure analysis of engineering parts. In the past decade, the study of fracture and failure analysis have made important contributions to the aviation, petrochemical and machine building industries. It also proves itself an indispensable means in uncovering criminal cases for the public security department.

1-6 稀土 - 过渡金属化合物

RARE-EARTH-TRANSITION METAL INTERMETALLIC COMPOUNDS

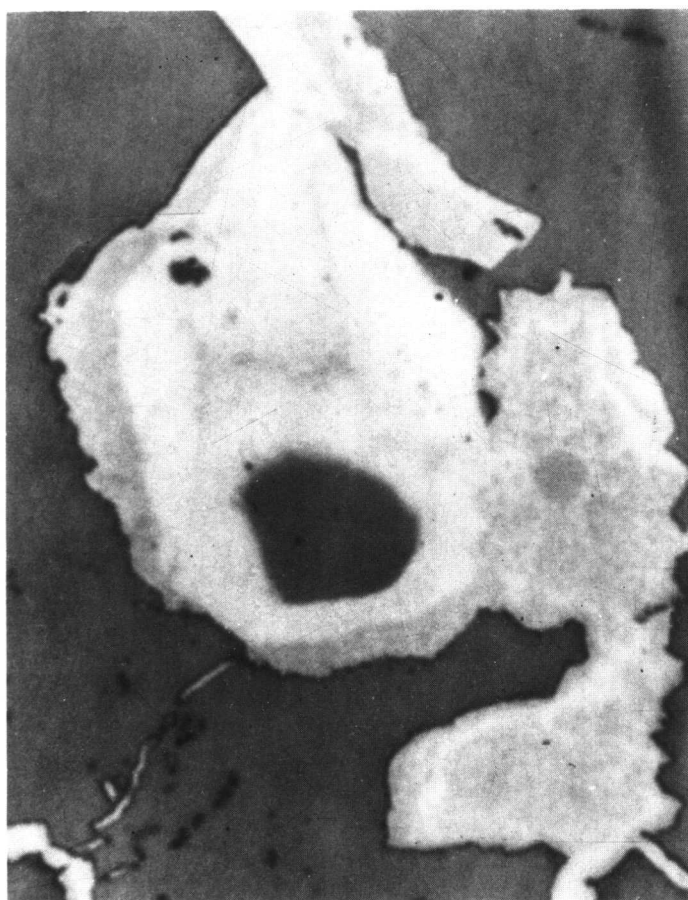


Fig. 1.6.1 $GdCo_5$ 720°C/48 days/ice-water quenched. Partially decomposed products morphologies (bright: Gd_2Co_{17} , grey: Gd_2Co_7).

我国有丰富的稀土资源，稀土的综合利用是金属研究所的重要方向之一。目前我所正研究发展功能更优越的稀土钴金属化合物。

在 $CaCu_5$ 型 RCO_5 化合物 ($R \equiv Pr, Gd, Y$) 中，以适量的其它金属元素 M 置换 Co 形成 $RCO_{5-x}M_x$ 化合物，它在 $650 \sim 800^\circ C$ ，仅局部分解为 R_2Co_{17} 与 R_2Co_7 或 R_2Co_{17} 与 R_5Co_{19} 相，一般以 R_2Co_{17} 相领先，分解机制为 Spinodal 型，以 Cu 或 Al 部分替代 Co ，在低浓度时分解为 R_2Co_{17} 和贫 Cu 或贫 Al 的 $R(Co, M)_5$ 相。

Th_2Ni_{17} 型 $Ho_2Co_{17-x}M_x$ ($M \equiv Ni, Cu$) 化合物居里温度 T_c 随成分的变化，出现最高值的异常现象，暗示着 Ni 和 Cu 在低浓度时对 Co 亚点阵 $Co-Co$ 交换作用的影响与高浓度时不同。目前正结合此现象进行 Mössbauer 谱的研究。

China is well known for her abundant rare earth resources. The comprehensive utilization of rare earth metals is one of the Institute's main research directions. Now we are studying and developing rare earth compounds with unique performances.

When the Co in the $CaCu_5$ type RCO_5 compound ($R \equiv Pr, Gd, Y$) is suitably substituted by a third metal M , $RCO_{5-x}M_x$ compounds with the same structure may be obtained. These pseudo-binary compounds will partially decompose spinodally into either R_2Co_{17} plus R_2Co_7 or R_2Co_{17} plus R_5Co_{19} after prolonged heating in the temperature range $650 \sim 800^\circ C$. (Fig. 1.6.1) When the substituent metal M is Cu or Al , the $RCO_{5-x}M_x$ compound partially decomposes spinodally into R_2Co_{17} and M -impolished $R(Co, M)_5$ at low concentration.

The Curie temperature of Th_2Ni_{17} type $Ho_2Co_{17-x}M_x$ ($M \equiv Ni, Cu$) compounds reaches a maximum at certain concentration of M which implies that the effect of these substituent metals on the $Co-Co$ exchange interaction of the Co -sublattice at low concentration may be different from that at high concentration. (Fig. 1.6.2) Now we are studying the Mössbauer spectrum of these compounds for this phenomenon.

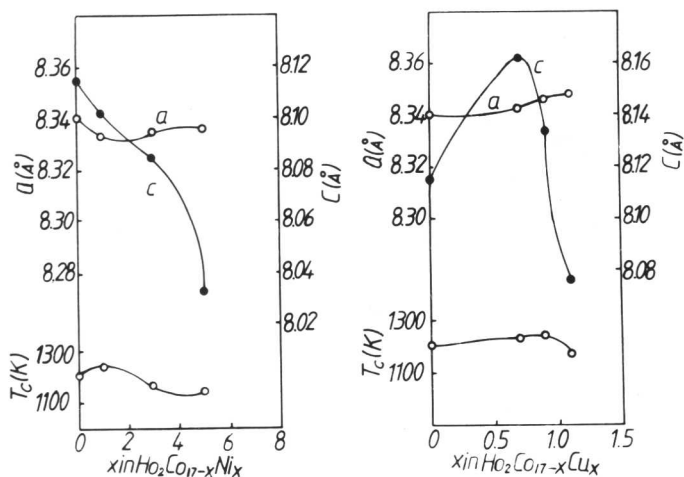


Fig. 1.6.2. Composition dependence of lattice constants and Curie temperature for $Ho_2Co_{17-x}Ni_x$ and $Ho_2Co_{17-x}Cu_x$ systems.

1-7 腐蚀、氧化与防护

CORROSION, OXIDATION AND PROTECTION

利用各种类型的自动微量热重分析仪，研究金属与合金的氧化动力学过程。着重研究稀土元素及稀土氧化物弥散质点改善镍基、钛基合金以及耐热钢高温氧化性能的机理。发现钇改善了 IN 939，钆改善了高温钛合金的抗氧化性能。

Investigation of oxidation kinetics of metals and alloys has been carried out with several type of automatic micro-thermogravimetry analysis systems. Present research work is to lay particular emphasis on the influence of rare earth elements and dispersed rare earth oxides particles on the mechanism of high temperature oxidation behaviour of nickel base alloys, titanium alloys, and heat-resistant steels. Yttrium and neodymium improve the oxidation resistance of IN939 alloy and high temperature titanium alloy, respectively.

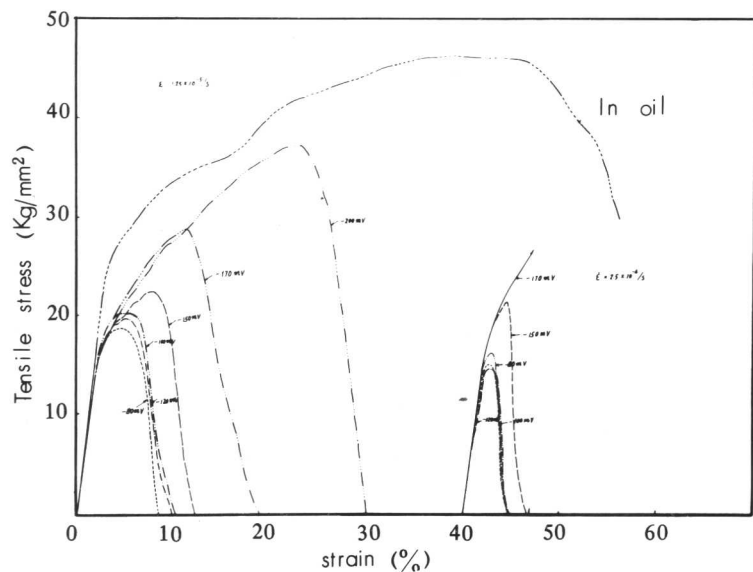


Fig. 1.7.3 Stress-strain curve of 18/8 type austenitic stainless steel in boiling 45% $MgCl_2$ under different potentials

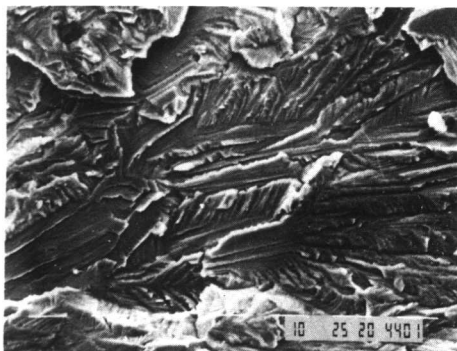


Fig. 1.7.4 Cleavage of 18/8 type austenitic stainless steel in boiling 45% $MgCl_2$ under different potentials.

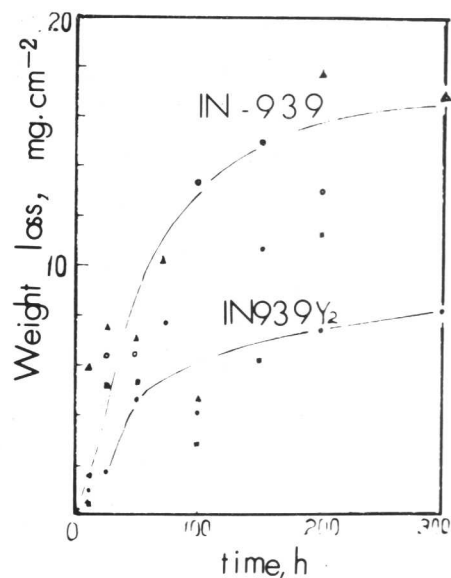


Fig. 1.7.1 Relationship between weight loss and time for IN939 alloy with and without Y

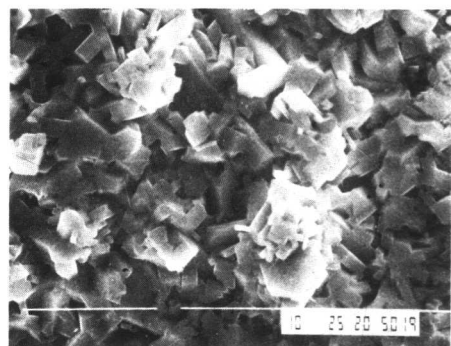


Fig. 1.7.2 Scanning electron microscope image of oxide scale of Ti alloy with Nd

利用恒变速率应力腐蚀试验机及电化学测试仪器研究金属及合金在某些特定环境介质下的应力腐蚀破裂行为。能够迅速可靠的评定出材料的抗应力腐蚀破裂的性能。

The behavior of stress corrosion cracking of metals and alloys under certain specific environments can be studied by using constant strain rate testing in conjunction with electrochemical measurements. This technique provides an effective, rapid and reliable laboratory method to assess susceptibility of materials to SCC.

1-8 焊接物理冶金

PHYSICAL METALLURGY OF WELDING

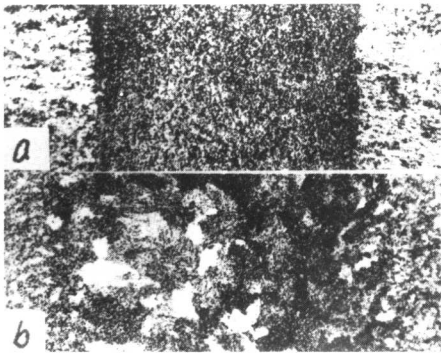


Fig. 1.8.1 Effect of elliptic scanning electron beam welding on the solidification of aluminium alloy weld.
 a. Scanning welding.
 b. Welding with out scanning.

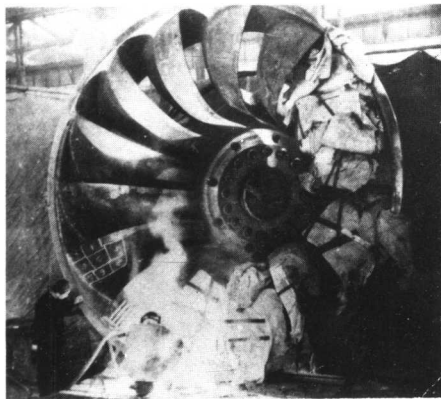


Fig. 1.8.2 44,000 Kws hydroturbine runner for Yan Guo Gorge Hydroelectric Power Station was surfacing welded with No. 1 hardfacing electrode at Harbin Electric Generator Manufacturing Plant.

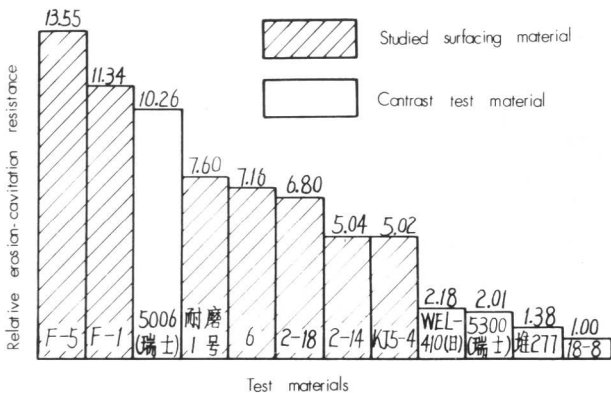


Fig. 1.8.3 Test results of relative erosion cavitation resistance of investigated surfacing materials

我所的焊接研究工作已有25年的历史。早在六十年代初期，就开始焊接材料和焊接物理冶金的研究，特别是对堆焊材料与堆焊层磨蚀机理，做了大量工作，不少成果已应用于生产。近几年来，又以经济建设为背景，开展了焊接接头强度与断裂、焊接热裂纹以及焊接氢致裂纹的研究，为改善电子束焊缝一次结晶，进行了焊接凝固结晶的研究。

Our institute has 25 years research history in the welding field. Since early sixties we began studying the physical metallurgy of welding and developing welding consumables, especially, great effort has been made in the area of surfacing consumables and the wear mechanism of surfacing layers. Many successful applications have been made in industry. In recent years, to meet the need of economic construction we engage in studying the strength and fracture of weld joints, hot cracking and hydrogen induced cracking during welding and solidification of the weld pool aiming at improving the microstructure of electron beam welds.

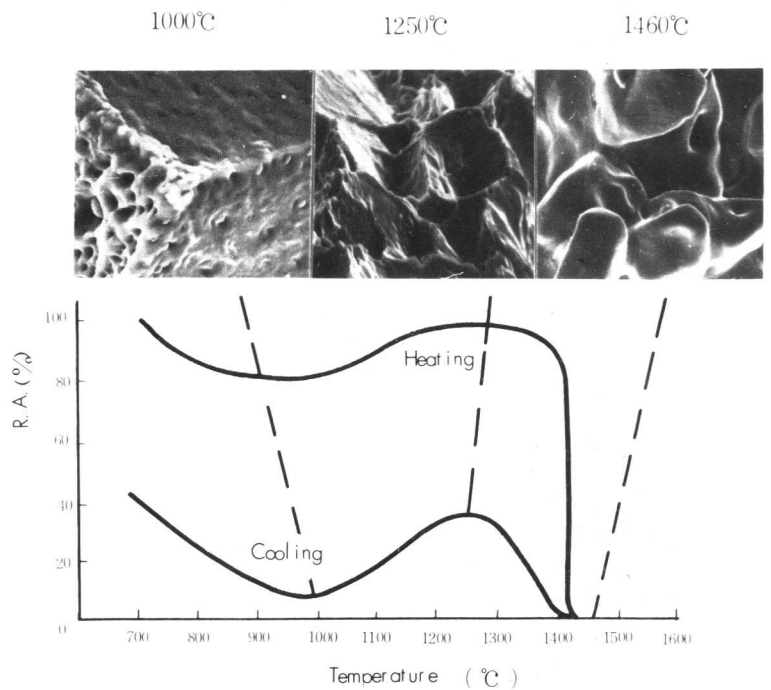


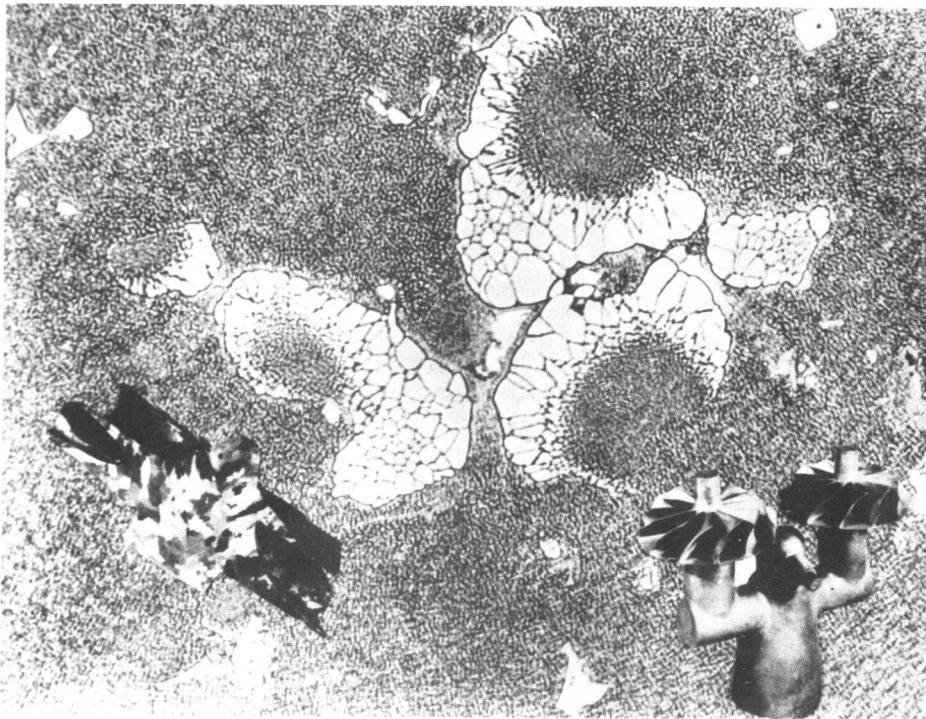
Fig. 1.8.4 Typical hot ductility curve and high temperature tensile fracture morphology of low carbon steel during welding

2 材料研究

MATERIALS RESEARCH

2-1 高温合金

SUPERALLOYS

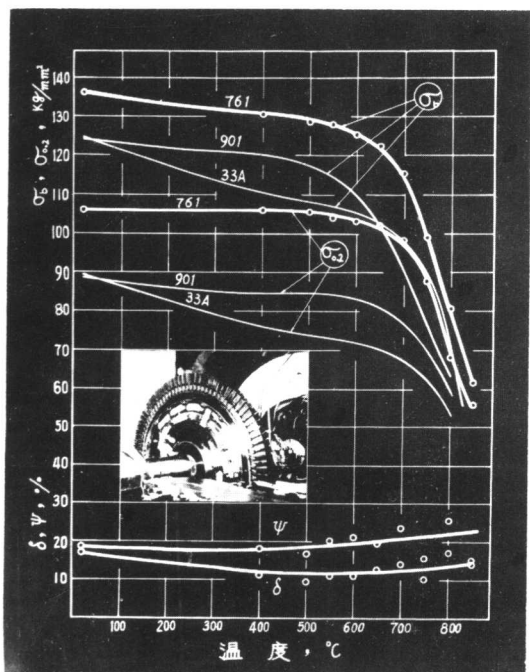


Microstructure of M17 cast superalloy

我所从五十年代中期以来，结合我国资源，发展了一系列的铸造高温合金和变形高温合金，应用于宇航、能源、化工和建材等方面。

近年来，正在深入研究金属的凝固原理、定向共晶、激光处理，以便改善现有高温合金性能及发展新型高温合金材料。

A series of cast and wrought superalloys based on national resources for aviation astronavigation, energy production, chemical industry and architectural engineering has been developed since the middle of fifties. Now the principle of solidification, directionally solidified eutectic alloys and laser treatment of alloy have been deeply investigated with the aim of improving the properties of superalloys in use and developing new alloys.



Mechanical properties of 761 Fe-base wrought superalloy in comparison with several turbine disc materials at different temperatures

2-2 钼合金

MOLYBDENUM ALLOYS

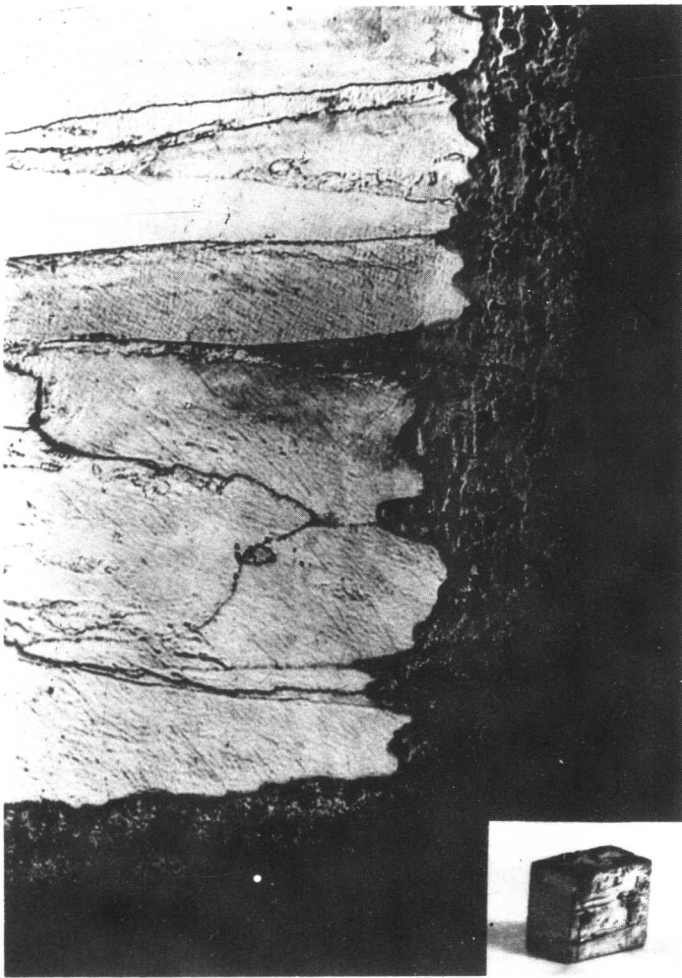


Fig. 2.2.1 Corrosion layer of Mo-30W plate after five months service in molten zinc.

我所研究钼合金已有二十多年历史，对多种钼合金的低温脆性、氧化机理以及防护、焊接、压力加工等工艺进行了一系列的研究。早年成功地将钼合金用作不锈钢穿管顶头、宇航工业用薄板等。近年来研制了高强度变形 Mo-30W 合金。除了良好的高温强度、硬度和一定的塑性外，它还兼有优异的抗熔融锌腐蚀能力。对其在锌液中的腐蚀行为进行了研究，并制成炼锌炉中的扬锌竖转轴，寿命已超过一年。解决了锌生产中耐蚀材料问题。

Investigation of molybdenum alloys has been carried out in this institute for more than twenty years. Mechanisms of low temperature embrittlement and high temperature oxidation-resistant coating, welding and working techniques, have been investigated extensively. Molybdenum alloy piercer for producing stainless steel tubes and molybdenum alloy sheets for the aerospace industry have been developed. Recently, a high strength wrought Mo-30W alloy was developed, which has excellent resistance to molten zinc corrosion and good ductility. Corrosion behavior of this alloy in molten zinc was studied. Mo-30W bars as vertical shafts in the zinc-smelting furnace has a service life longer than one year which satisfies the requirements demanded by the zinc industry.

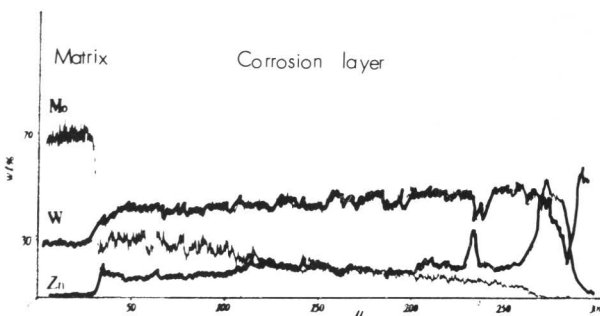


Fig. 2.2.2 Concentration profile of Mo, W and Zn in corrosion layer of Mo-30W alloy

2-3 钛合金

TITANIUM ALLOYS

根据国家新材料、新技术发展需要,开展高温钛合金、形状记忆合金、耐蚀钛合金及汽轮机长叶片材料研制和应用研究。

相变是发展和应用钛合金的重要研究课题。根据电子浓度理论,研究有序相 Ti_3X (Al, Ga, Sn) 形成与过渡族元素 Sc, Zr, V, Nb, Mo, 及间隙原子 O, C, N 电子结构的关系。用电子显微镜暗场技术,确定 Ti_3X 相析出相界和特征电子浓度值,建立相计算模型,并用于高温钛合金热稳定性控制和合金设计。

另外,采用唯象理论和计算机方法,配合电镜技术,研究热弹性马氏体相变和形状记忆效应。

研究钛加入 Mo, Nb, W, Ta 以及 Pd 和 Y 等元素在盐酸介质中的腐蚀与钝化行为。采用 ESCA 研究钝化膜的组成及其对腐蚀稳定性的影响。

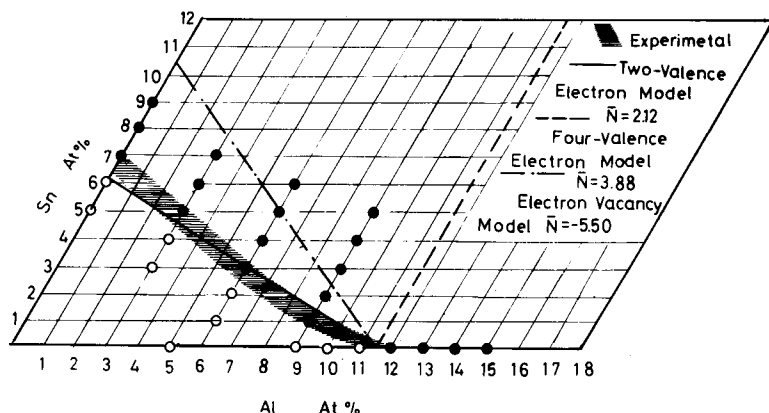


Fig. 2.3.1. Determination of phase boundary of α/Ti_3 (Al, Sn) and characteristic electron concentration
Valence electron of alloying elements: $N_{Ti}=2$, $N_{Al}=3$ (S^2P^1), and $N_{Sn}=4$ (S^2P^2)

Based on the need of new materials and techniques, research on titanium alloys and their applications has been carried out. It includes high temperature alloys, shape memory alloys, corrosion-resistant titanium alloys, steam turbine blades etc.

Phase transformation study is of vital importance in the development and application of titanium alloys. According to the electron concentration concept, the correlations between formation of ordered Ti_3X phase ($X=Al, Ga, Sn$) and electronic structure of alloying elements, including transition metals Sc, Zr, V, Nb, Mo, and interstitial elements O, C, N, have been investigated. In this process, the α/Ti_3X phase boundaries have been determined by using the EM dark-field technique. As a result, a characteristic electron concentration criterion is proposed and a phase calculation model established. This model can be used in controlling thermal stability of high temperature titanium alloys and is conducive to the designing of alloys.

In addition, the phase transformations of thermoelastic martensite, and the shape memory effects are being investigated by phenomenological theory and computer program, in conjunction with EM technique.

On the other hand, the effect of alloying elements, Mo, Nb, W, Ta, Pd and Y etc., on the corrosion and passivation behaviour of titanium alloys has been investigated in solutions containing HCl. The composition of passive film and its effect on the corrosion resistance stability are studied by ESCA.

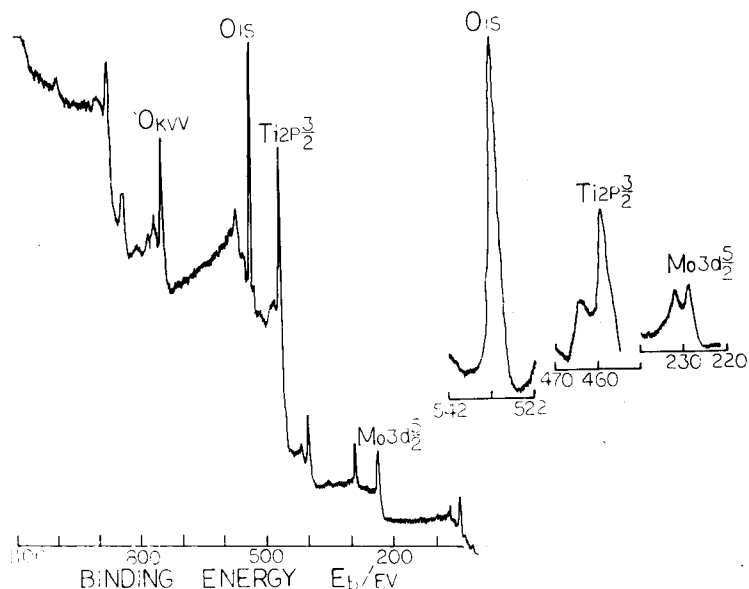


Fig. 2.3.2. ESCA analysis of passive film on Ti-Mo alloy in HCl- CH_3OH solution

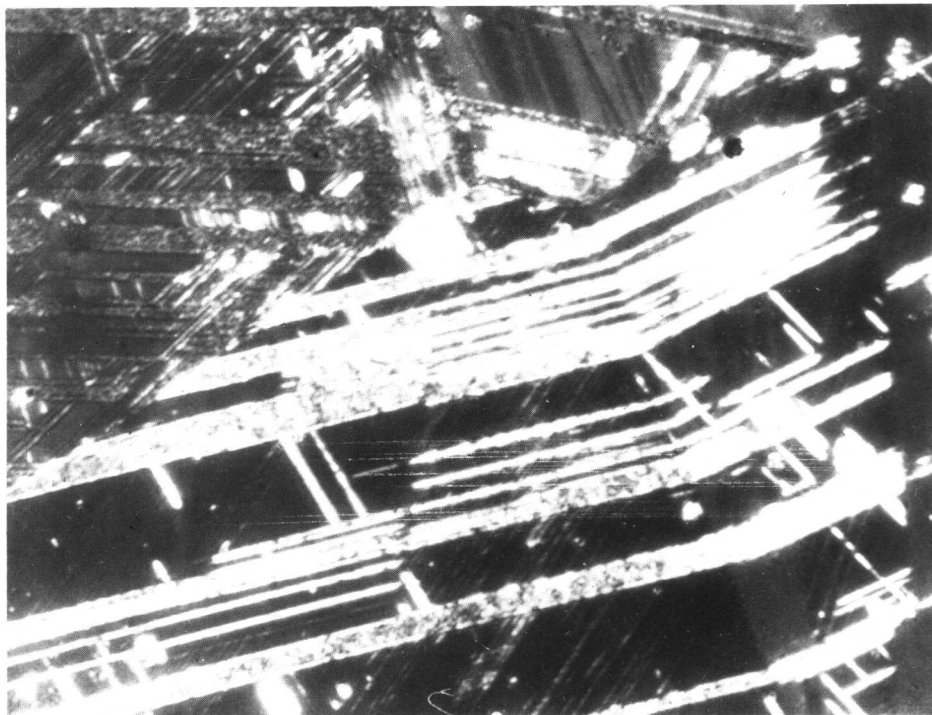
2-4 低温钢

CRYOGENIC STEELS

我们曾发现铝能强烈抑制铁锰合金中 $\gamma-\epsilon$ 相变(见右图),由此首创出一种超低温无磁钢-15Mn26Al4。它在300-20K间具有稳定的奥氏体组织和优良的力学及物理性能。以后又先后研制了一系列低温钢,建立了干涉层金相等特种金相方法,发展成一个国内领先的低温钢研究集体。

研究方向:

- 研究低温相变的热力学、动力学及合金在低温下的组织结构,以了解合金成份、温度与组织之间关系的规律。
- 通过对低温力学性能、物理性能及组织结构的深入研究,了解合金组织与性能之间关系的规律。
- 设计、研制和发展新型低温材料。
- 发展相应的低温实验技术。



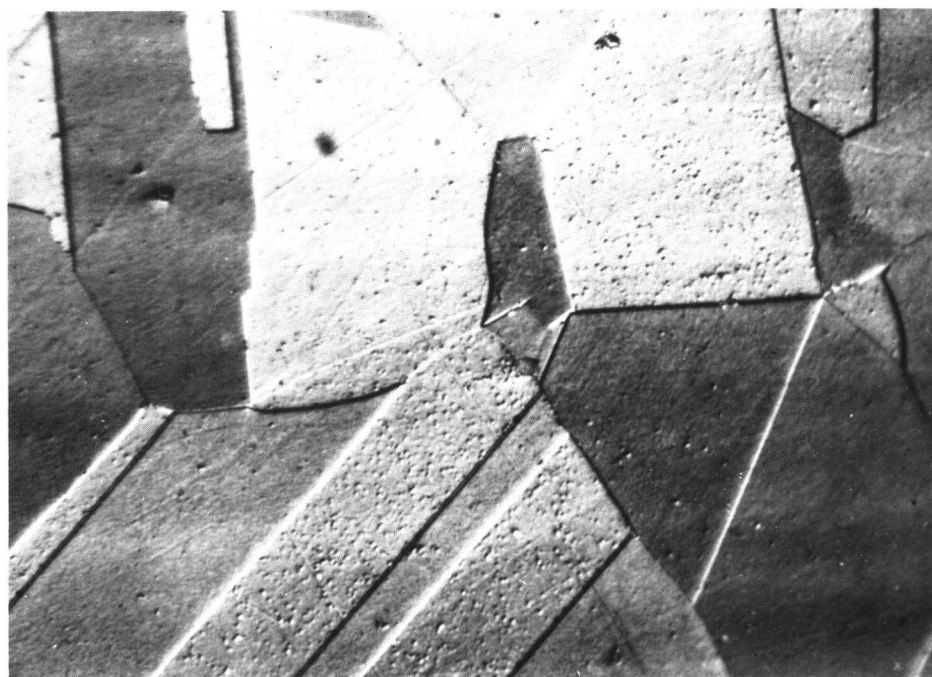
15Mn26

$\gamma+\epsilon$

It is found that aluminium markedly suppresses the $\gamma - \epsilon$ martensite transformation in Fe-Mn alloys (see figures on right). A cryogenic non-magnetic steel-15Mn26Al4 has been developed which has stable austenitic structure as well as excellent mechanical and physical properties in the temperature range 300 to 20K. A series of cryogenic steels has been developed and special metallographic methods, e.g. interference layer method have been established.

Scope of research:

- thermodynamics and kinetics of phase transformations, and structure of alloys at cryogenic temperatures
- properties and structures of alloys
- design, investigation and development of new cryogenic materials
- development of relevant cryogenic experimental techniques.



15Mn26 Al4

γ