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纳米硬度技术在表面工程力学性能检测中的应用

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摘要:结合纳米硬度技术测量各类薄膜和块体材料表层的纳米压痕硬度、弹性模量、断裂韧性、膜厚、微结构的弯曲变形,采用纳米划痕硬度技术测量各类薄膜和块体材料的粗糙度、临界附着力、摩擦系数、划痕横剖面。纳米硬度计是检测材料表层微米乃至几十纳米力学性能的先进仪器,可广泛应用于表面工程中的质量检测。

关键词:纳米压痕硬度;纳米划痕硬度;力学性能;表面工程

中图分类号:TG174.444;O484　　**文献标识码**:A

随着气相沉积技术的快速发展和广泛应用,传统的机械方法对几个微米乃至几个纳米膜层的力学性能表征已无能为力。纳米硬度技术(包括纳米压痕法和划痕法)正成为表面工程力学性能检测中的重要手段[1]。

近20年来,纳米硬度技术发展较快,已有多家生产商研制出商品化的仪器[2]。现以我们采用MTS Nano Indenter XP系统的实验结果为例,说明它在表面工程中的应用。

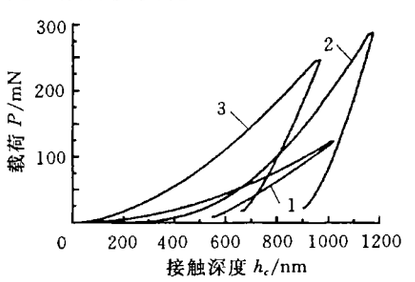
1　纳米压痕技术的应用

纳米压痕硬度计是一种先进的能提供高分辨率连续载荷和位移测量的材料表面(深度10^3nm～10^1nm)力学性能测试仪器。它明显拓宽了传统显微硬度的能力,可完成多种力学性能的测试,如硬度、弹性模量、断裂韧性、膜厚、微结构的弯曲变形。对不会导致压痕周围凸起(pile—up)的材料,如大多数陶瓷、硬金属和加工硬化的软金属,纳米压痕硬度和弹性模量的测量精度通常优于10%[2,3]。

1.1　纳米压痕硬度和弹性模量

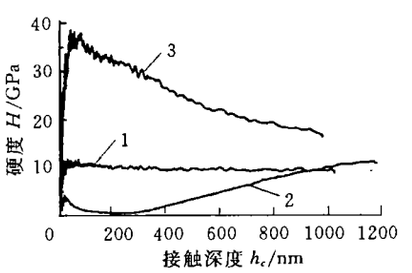
纳米压痕硬度与显微压痕硬度的定义和工作方式不同。纳米压痕硬度的定义为在压入过程中压痕表面积投影上单位面积所承受的载荷,它反映样品承受接触载荷的能力。而维氏硬度为残余压痕表面积上单位面积所承受的载荷,它反映样品抵抗残余变形的能力。纳米压痕技术是从连续压入深度测量中直接计算接触面积,而显微硬度通过测量卸载后的压痕对角线再查表得压痕表面积。

为了便于比较,首先提供了该仪器标准试样熔融硅(fused silicon)的压痕实验结果。熔融硅的优点主要是表面光滑、抗氧化、非晶、各向同性、无加工硬化、中等范围的力学特性,典型的陶瓷行为、在卸载时有较大的弹性恢复、无明显时间相关性。图1反映了压针在试样中的加卸载过程,卸载曲线的末端基本上反映卸载时的残余压痕深度。从图2和图3中可以看出,熔融硅的硬度和弹性模量不随压痕深度变化。



1.熔融硅　2.钛膜/氮化硅基底　3.TiN膜/9Cr18钢基底

图1　加卸载曲线



1.熔融硅　2.钛膜/氮化硅基底　3.TiN膜/9Cr18钢基底

图2　硬度随压痕接触深度变化曲线

使用离子溅射技术,在氮化硅上沉积厚约1.00 μm的钛膜。按显微硬度的经验,压痕深度为

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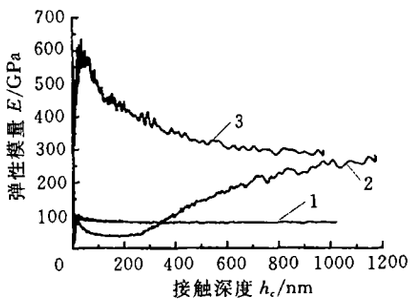

1. 熔融硅　2. 钛膜/氮化硅基底　3. TiN 膜/9Cr18 钢基底

图 3　弹性模量随压痕接触深度变化曲线

1/10～1/5 膜厚时，基底对膜的力学性能测试结果无影响[4]。从图 2 和图 3 中可以看出，在压痕深度约在 0.05～0.30 μm 范围内，硬度和弹性模量保持稳定，这是薄膜的力学性质。随着压痕深度的增加，硬度和弹性模量不断增大，这是基底对薄膜性质的影响所致。当压痕深度超过 1.00 μm 时，硬度和弹性模量随压痕深度增加的趋势变缓，逐渐接近基底性质。这反映了软膜硬基底材料的力学性能变化规律。同时，可从硬度和弹性模量随深度变化曲线中粗略估计膜厚。

采用等离子电弧沉积技术，在 9Cr18 钢上沉积厚约 0.50 μm 的 TiN 膜。从图 2 和图 3 中可以看出，50～100 nm(1/10～1/5 膜厚)时，TiN 膜的硬度和模量分别为 35GPa、550GPa。随着压痕深度的增加，TiN/9Cr18 的硬度和模量逐渐变小，并接近于基底 9Cr18 的性质，如弹性模量趋于 200GPa 即钢的弹性模量，说明基底的影响逐渐变大。这反映了硬膜软基底材料的力学性能随压痕深度的变化趋势。

由上可知，该技术能检测出硬度和弹性模量随压痕深度变化的规律，是研究薄膜界面力学的有效手段。

1.2　断裂韧性[5～7]

用 Berkovich 压头获得材料的硬度 H 和弹性模量 E，然后再用 Cube－corner 压头在材料中产生径向裂纹，见图 4。断裂韧性和径向裂纹长度的关系为

$$K_c = \alpha\left(\frac{E}{H}\right)^{1/2}\left(\frac{P}{C^{3/2}}\right) \qquad (1)$$

式中，P 为作用在 Cube－corner 压头上的最大载荷；C 为径向裂纹长度；α 为与压头形状相关的经验系数。

由于 Cube－corner 压头要比 Berkovich 压头尖得多[2]，易于在压头周围材料中产生较大的应力和应变，便于压痕裂纹的形成和扩展。在较小载荷作用下，Cube－corner 压头可以在许多脆性材料中产生带有径向裂纹的亚微米压痕，特别适合于检测薄膜或材料微小区域的断裂韧性。

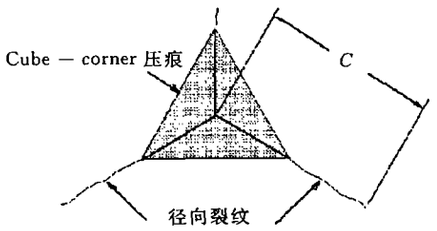

图 4　Cube－corner 压头产生的径向裂纹

1.3　厚度

在 GT35 钢上沉积 DLC 膜，按沉积速率估计膜厚约为 500nm。从图 5 中可以看出，在压痕深度为 620～720 nm 时加载曲线上有一平台。对一般材料来说，加卸载曲线多为连续变化的。经分析可知，该平台对应于膜厚，是由膜的粘着失效(adhesion failure)造成的。这可以作为一种测膜厚的方法，该方法仅对性质差别明显的硬膜软基底适用。

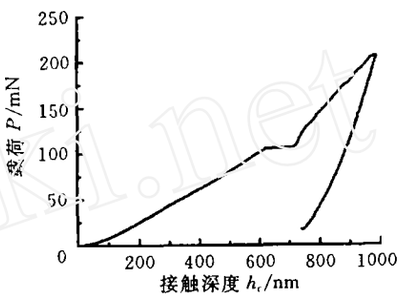

图 5　DLC 膜厚的测量

1.4　微结构的挠度

在硅片上电镀镍膜，采用微加工工艺将膜制作成 100 μm×18 μm×3.2 μm 的微桥。压针为楔形(wedge tip)，楔长 28 μm，楔角 45°。为研究镍微桥的弹性弯曲行为，压针楔长沿微桥宽度方向作用在微桥的中间，使用三次加卸载的方法测其挠度，见图 6。

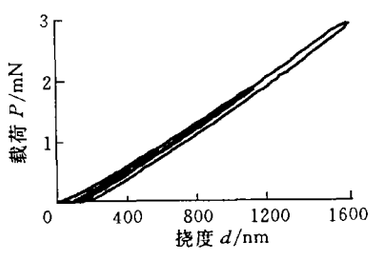

图 6　镍微桥的弯曲

2　纳米划痕技术的应用

在表面工程中，需要了解材料表面的变形机理和抗磨损性能。划痕实验过程包含了许多变形和破坏过程。目前，纳米划痕硬度技术能定量分析

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材料表面的划痕变形机理和摩擦行为。同以前的划痕计相比，载荷和位移的分辨率提高，改进对临界载荷的确定方式[4]。

2.1 粗糙度、临界载荷和摩擦系数

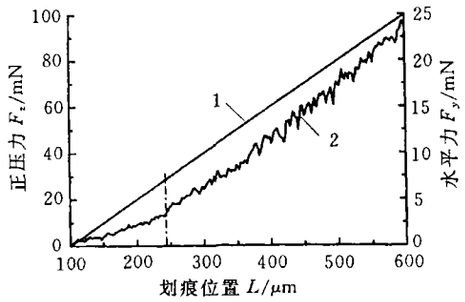
采用等离子电弧沉积技术，在 9Cr18 钢上沉积 TiN 膜。图 7 为 TiN/9Cr18 的纳米划痕实验结果。实验主要分三步：①预扫描，作用在压针上的正压力为 20 μN，样品台以 10 μm/s 的速度移动，测量出试样表面的形貌或粗糙度。TiN 薄膜表面比较光滑，在 100nm 内波动，见图 7b。②刻划过程，正压力线性增加，最大值为 100mN，水平力随正压力的增加变化较大，见图 7a，划痕深度和位置的关系见图 7b。在初始阶段，随着正压力的增加，划痕深度也近似线性增加。当深度增至－140 nm 对应位置为 240 μm 时，划痕曲线明显变化。对应的摩擦系数也明显增加，说明压针划到基底了，对应的正压力和水平力分别为 28.2mN、4.2mN，该力定义为薄膜和基底之间发生剥离的临界载荷[4]。③后扫描，正压力仍为 20 μN，测量出卸载后残余划痕的形貌，即表面损坏信息。由于残余划痕深度明显小于划痕深度，说明 TiN 膜具有较好的弹性恢复能力。图 7c 中，在 140～240 μm 范围内的摩擦系数较稳定（约 0.15），为压针在 TiN 中刻划的摩擦系数。随后，摩擦系数逐渐变大，说明压针在基底中越划越深，最终将接近基底的摩擦系数。

2.2 划痕横剖面

另一种划痕方法主要通过测划痕横剖面（cross－profilometry）的形状来研究粘塑性材料的松弛（visco－plastic relaxation）性能，特别适合于研究受时间和温度影响的高聚物的粘塑性松弛性能。目前，该技术已成为评价汽车喷漆质量的重要手段[8]。实验是在单晶铝上进行的，过程如下：①预扫描，垂直作用在试样上的力为 50 μN；主要测量试样表面的粗糙度。②刻划，垂直作用在试样上的力为 0～40 mN 连续增加，断裂时的临界载荷和压痕深度为分别为 8.76 mN 和－2313 nm；③后扫描，垂直作用在试样上的力为 50 μm；主要测量残余划痕深度，见图 8。①横扫描，最后在划痕位置 750 μm 处用 5 mN 的力，沿横剖面扫描，见图 9。

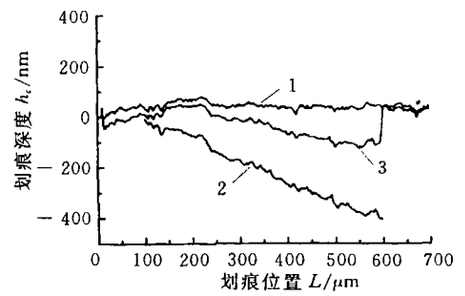
3 结束语

目前，纳米硬度技术已广泛应用于表面工程中的力学性能检测。该技术经十多年的发展，取得了长足的进步。



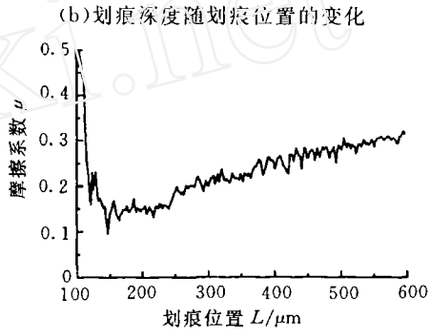
1. 正压力　2. 水平力

(a)正压力和水平力随划痕位置的变化



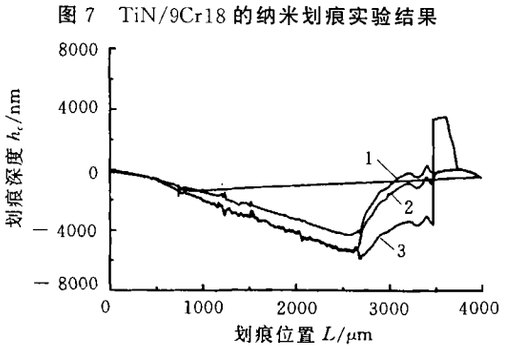
1. 预扫描　2. 刻划　3. 后扫描

(b)划痕深度随划痕位置的变化



(c)摩擦系数随划痕位置的变化

图 7　TiN/9Cr18 的纳米划痕实验结果



1. 预扫描　2. 刻划　3. 后扫描

图 8　铝中的划痕过程

(1)结合新观察技术，如用原子力显微镜观察纳米尺度的压痕和凸起形貌。

(2)发展新测量技术，如 DCM 技术的发展，拓宽了测量高聚物粘弹性能的频率范围，提高了测量纳米薄膜特性的能力。

(3)建立新分析技术，有限元模拟已成为有力的分析技术。

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高速机构梁类弹性构件声辐射分析与计算

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摘要:以四连杆机构的弹性连杆为研究对象,采用运动弹性动力学分析,求取振体表面法向速度;对其运动过程中的声辐射功率及结构模态声辐射效率进行了预测,为梁类高速运动弹性构件的声辐射控制提供了理论依据。

关键词:弹性构件;运动弹性动力学分析;声辐射;噪声

中图分类号:TH133.5;TB53　　**文献标识码**:A

目前许多机电系统正向复杂化、高速化及相对重载化的方向发展,但随之而来的噪声问题也日益突出。弹性构件在现代机器中得到了普遍应用[1,2],但在高速工作过程中的声辐射不容忽视。本文以具有广泛代表性的做平面运动的弹性梁为研究对象,对梁类弹性构件高速运动的声辐射问题进行了讨论。

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1　振动体表面法向速度的求解

对于做平面运动的梁,整体的刚体运动与本身的弹性振动存在耦合作用[3,4]。所以在进行动态特性分析时,应当采用运动弹性动力学方法。这里以常见的曲柄摇杆机构中的连杆为研究对象,见图1。本文假

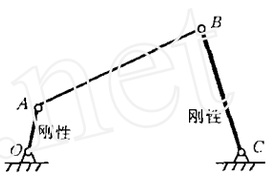

图1　曲柄摇杆机构

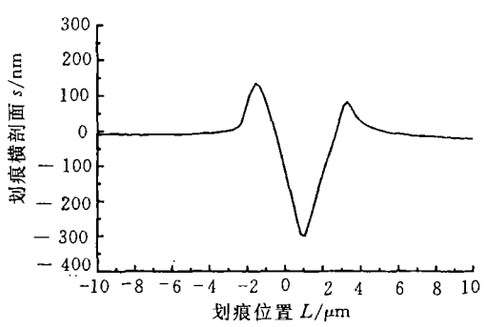

图9　铝在750 μm处的划痕横剖面

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discuss its motion stability.

Key words: passive magnetic bearing equivalent time-varying magnetic field conductor loop stability electrodynamic suspension

Research on the Machine Loading Planning in Flexible Manufacturing System YANG Honghong (Shanghai Jiaotong University, Shanghai, China) WU Zhiming p 2137-2141

Abstract: The machine-loading problem in a flexible manufacturing system is considered in this paper. The loading problem is defined as the allocation of part and required tools to the machine during operation, so as to optimize the objective function subject to some constraints. The problem is formulated as integer programming. A Genetic Algorithm (GA) is employed to solve this problem. Computational results are presented to demonstrate the effectiveness of the algorithm. The proposed approach can be extended to large-scale machine loading problems.

Key words: FMS production planning machine loading genetic algorithm

Reserach on Inventory Control Model Based on MRP Ⅱ SONG Guofang (Shanghai University, Shanghai, China) YU Jie QI Ershi p 2141-2144

Abstract: In order to fetch up the shortages of inventory management in MRP Ⅱ system, using statistics an inventory control model based on MRP Ⅱ is put forward. This model can use MPS and BOM file in MRP Ⅱ to determine the final material quantity and lead-time, and it also can use statistics method to determine the most reasonable order batch and time. This model can reduce inventory fare and enhance enterprises' inventory management efficiently, which is proven by practice, and will provide reference for enterprises.

Key words: MRP Ⅱ inventory control JIT turnover

Multi-software collaboration Cutting-stock Method Based on ASP and Its Implementing Technologies YAN Chunping (Chongqing University, Chongqing, China) LIU Fei LIU Ying ZENG Yudan p 2144-2147

Abstract: Cutting-stock is a NP complicated problem. Each of optimum software can obtain the cutting results by using proximate and heuristic methods. But different optimum methods and their optimum software may not obtain perfect results according to certain data framework. Furthermore, enterprises may not buy lots of optimum software to solve the problems. Aiming at the problems, a multi-software collaboration cutting-stock method based on ASP is proposed, and corresponding implementing technologies based on agent are given. From experiments, the results show that this method promotes the general optimum results of cutting-stock greatly.

Key words: cutting-stock application service provider (ASP) agent collaboration solving

Nano-hardness Techniques and Its Applications in Mechanical Property Measurements for Surface Engineering ZHANG Taihua (Chinese Academy of Sciences, Beijing, China) YANG Yemin p 2148-2151

Abstract: The rapid developments of surface engineering make progress in the measuring techniques for mechanical properties. This paper is specially focused on introducing measuring ways and results of Nano Indenter XP system made in MTS, such as elastic modulus, hardness, fracture toughness, film thickness, microbeam and microbridge deflection, surface roughness, critical load, friction coefficient, cross-profilometry. Nano-hardness techniques are the advanced ways for measuring mechanical properties of material surfaces from 103nm down to 101nm. So, it may be widely applied to quality assessment in the field of surface engineering.

Key words: nanoindentation nanoscratch mechanical property surface engineering

Analysis and Calculation of Acoustic Radiation of a Beam-like Flexible Components in High-speed Mechanisms LU Jianwei (Shantou University, Shantou, Guangdong, China) ZHANG Xianmin SHEN Yunwen p 2151-2154

Abstract: Based on the kineto-elastodynamics analysis of a flexible linkage in four-bar mechanism, the surface normal velocity of the linkage is obtained. In addition, the acoustic radiation power and radiation efficiency of the beam is predicted, which provides theoretical basis for the acoustic radiation control of high-speed flexible beam-like components.

Key words: flexible components kineto-elastodynamics analysis acoustic radiation noise

Research on Depositing C_3N_4 Superhard Coating Film onto HSS Tool WU Dawei (Wuhan University, Wuhan, China) WU Yuexia PENG Yougui FAN Xiangjun p 2154-2157

Abstract: For depositing advanced superhard coating film of Carbon Nitride onto high speed steel tool, by combination of DC reaction magretron sputtering with multiple arc-plating, the superhard coating is deposited onto high speed steel, and a compound thin film exists in the form of hard crystalline phase using X-ray diffraction (XRD) analysis. The microhardness Hv of the compound film is of 50.5～54.1GPa. The adhersive force of superhard coating film with high speed steel base is of $L_c = 40$～80N, by scratch test to measure the critical load. Many kinds of tool proves that coating has a very high wearabil ity, the tool life is great improved, as compared with TiN coated cutting tools and uncoated tools.

Key words: carbon nitride superhard thin film high speed steel tool (HSS) coated cutting tool

Development Trend of High-speed Train and Their Vibration Suppress System PAN Shuangxia (Zhejiang University, Hangzhou, China) YANG Likang FENG Peien p 2157-2160

Abstract: Facing the trend to increase the train speed, the character istics and feasibility of tilting train, wheel/rail train and maglev train are compared. The vehicle suspension system is especially focused on in this paper because it is very important to increase train speed and good comfort quality. The suspension system evolution of passive mode, active mode and semi-active mode is studied. Their advantages and disadvantages are analyzed based on the basic structure and function. Furthermore, the effective control strategy for active and semi-active suspension is investigated according to the complex non-linear system control law. It is concluded that smart semi-active damper, effective and reliable controller, mechatronics modeling and security should be researched.

Key words: high-speed train vehicle suspension system suspension mode vibration control

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